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Overburden materials from surface coal mines in southwestern Indiana were analyzed for physical and chemical properties. Plant growth potential of selected materials, with and without sewage sludge and fertilizer amendments, was evaluated in greenhouse pot culture and outdoor containers using alfalfa, small grains and tree seedlings.

The general ranking of overburden materials for plant growth was lacustrine sediment > A horizons > B horizons = glacial tills = loess > brown shale > sandstone > gray shale > black fissile shale. Amendments ranked sewage sludge > fertilizer > as surface plant growth media. The B horizons, partially weathered loess and glacial tills were similar in productivity and are suitable for use in the plant root zone. The sandstone and brown shale are not suitable in the upper root zone due to undesired physical properties or content of phytotoxic elements in sandstone. The gray and black fissile shales are unsuitable for plant growth and should be avoided in the rooting zone.

Regression analysis did not reveal overburden properties that could be used consistently for predicting plant growth potential. Electric conductivity of the material extract and water storage capacity were most frequently significantly related to growth.

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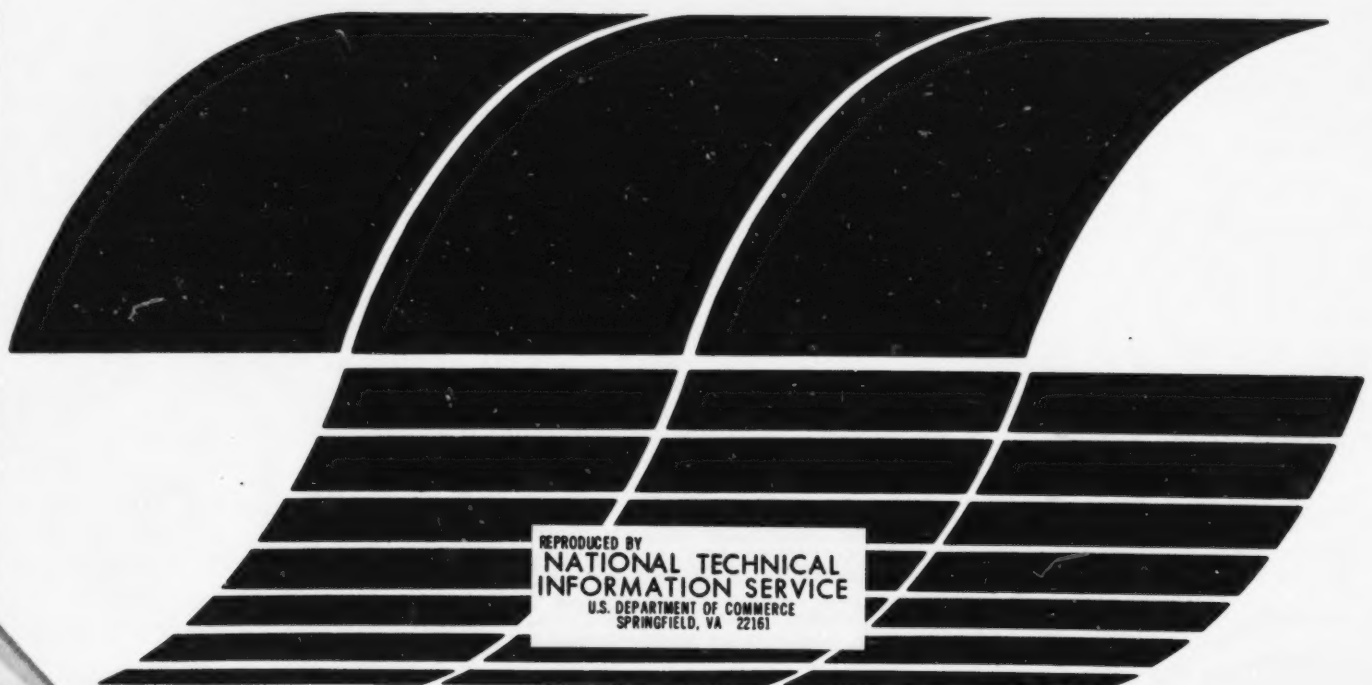

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The views and conclusions contained in this report are those of the authors and should not be interpreted as representing the official policies or recommendations of the Science and Education Administration, Cooperative Research, U. S. Department of Agriculture.

FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory-Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

Reported here is the results of a study supported cooperatively by U.S. Department of Agriculture and U.S. Environmental Protection Agency at Purdue University. The project was a part of the Interagency Energy-Environment Research and Development Program. The results of this study should be of interest to these persons engaged in the planning and reclamation of surface mined lands, because properties of overburden materials that may serve as predictors of potential plant growth have been evaluated.

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ABSTRACT

This investigation was conducted to determine physical and chemical properties of mineland overburden, to evaluate plant growth in these materials, and to identify properties of overburden materials before mining that may serve as predictors of potential plant growth.

Eighteen overburden materials from 5 surface coal mines in the Illinois coal basin of southwestern Indiana were sampled and analyzed for 20 physical and chemical properties. Twelve are unconsolidated materials, including A and B horizons, loess, lacustrine sediments, and glacial tills, and six are rock strata that break and weather easily. Growth potential of overburden materials, with and without sewage sludge and fertilizer amendments, were evaluated in greenhouse pot culture using alfalfa, wheat and white pine. Oats yield, and survival and growth of Virginia pine and yellow-poplar were evaluted for 10 materials in outdoor containers.

Regression analysis of plant growth against chemical and physical properties of the overburden materials did not reveal properties that could be consistently used in a formula approach to predicting plant growth material. Electrical conductivity of the material extract and water storage capacity were most frequently significantly related to growth.

The general ranking of overburden materials evaluated for plant growth potential was lacustrine sediment $\geq$ A horizons > B horizons = glacial tills = loess $\geq$ brown shale > sandstone > gray shale > black fissile shales. Lacustrine sediment and A horizons were clearly superior materials for use as surface plant growth media. The B horizons, partially weathered loess and glacial tills were similar in productivity and are suitable for use in the plant root zone. With amendments, B horizon and loess may be acceptable for surface use, but are not likely to equal A horizon material in productivity. The sandstone and brown shale are not suitable in the upper plant root zone due to undesired physical properties and potentially phytotoxic elements in sandstone. The gray and black fissile shales used are unsuitable for plant growth and should be buried to avoid direct contact with plant roots and to reduce subsequent oxidation and leaching.

Addition of sewage sludge resulted in vastly improved growth of wheat and to a lesser extent alfalfa on most materials in the greenhouse. Fertilization with N-P-K was less effective than sludge but usually produced increased plant growth. Amendment of the upper rooting zone with major nutrient elements and organic matter from wastes such as sludge would be a beneficial practice.

This report was submitted in fulfillment of Contract No. 684-15-18 by Purdue University in cooperation with the Science and Education Administration, Cooperative Research Unit, USDA, under the sponsorship of the U.S. Environmental Protection Agency. This report covers the period from July 21, 1976 to September 30, 1978 and was completed as of April 15, 1979.

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Overall administration of the project was coordinated by Dr. B. J. Liska, Director, Indiana Agricultural Experiment Station, and Dr. Eilif V. Miller, Science and Education Administration, Cooperative Research, U.S. Department of Agriculture.

INTRODUCTION

With increasing demand for coal as an energy source, it is evident that coal-mining activities will expand significantly in the United States. A major proportion of coal extraction in the foreseeable future will be by surface-mining which is more disruptive to land areas than underground mining.

The Illinois Coal Basin is a major energy resource with coal reserves estimated at 114 to 136 billion metric tons; more than 16 billion metric tons of recoverable coal occurs in southwestern Indiana. Land disturbance from surface coal-mining in Indiana alone exceeds 1800 ha per year for a total of about 62,500 ha to date.

Although coal production is a necessity, the control of environmental problems created by surface-mining and restoration of disturbed land to productive use are also important. These aspects were emphasized in federal legislation regulating surface-mining and reclamation (PL 95-87) enacted in 1977. In mineland reclamation, the objective is to leave a valuable resource, i.e., land areas of the highest possible value following coal extraction. One of the major problems of reclamation, however, involves requirements and related criteria for cast overburden disposition to provide the best plant growth media. The approach generally used is merely to require that original "top soil" be returned to the cast overburden surface. Although this may be satisfactory, other overburden strata may be adequate for plant growth and could be placed near the surface more efficiently and economically. Analysis of undisturbed overburden to determine quantity, layer thickness and volume, and quality, physical and chemical properties, of various soil and geologic strata above the coal seam may be a useful technique in assessment of its potential for plant growth. Such analyses would also identify location and amounts of phytotoxic and pollutant materials that should be avoided in the surface plant growth zone.

The objectives of this investigation were: (1) to determine physical and chemical properties of undisturbed mineland overburden that predict the suitability of various strata for plant growth; (2) to evaluate by bioassay the potential of overburden materials as plant growth media with and without amendments; and (3) to develop criteria based on these tests that can be used prior to mining to determine which materials would be acceptable for placement on the surface. Analyses were made on selected overburden materials extracted from active coal mine highwalls in glaciated and unglaciated areas of southwestern Indiana. Plant growth potential of overburden materials was evaluated for selected crop and tree species in greenhouse pot studies and large outdoor containers. Fertilizer and sewage sludge overburden amendments were incorporated in the greenhouse plant growth studies.

CONCLUSIONS

The general ranking of overburden materials for plant growth potential was lacustrine sediment ≥ A horizons > B horizons = glacial tills = loess ≥ brown shale > sandstone > gray shale > black fissile shales. This ranking is based on evaluation of physical and chemical properties, seed germination and emergence, plant growth and yield, and uptake of nutrients and toxic elements.

Lacustrine sediment and A horizons were clearly superior materials for use as surface plant growth media. These materials exhibited favorable physical and chemical properties and consistently produced the best growth of alfalfa, cereal grains and tree seedlings in greenhouse or outdoor container experiments. Also, they contain an adequate supply of most plant nutrients, except phosphorus.

The B horizons, partially weathered loess and glacial till materials were essentially similar in plant productivity and are suitable alone or in mixtures for use in the plant root zone. B horizons and loess possess physical characteristics such as density, texture and water-holding capacity within the desired range and do not exhibit toxic chemical properties. With organic amendments such as sewage sludge and fertilization, these materials may be a suitable alternative for use on the surface of reconstructed mineland soil, but are not likely to equal A horizon material in productivity. Further, the thickness of B horizon and loess as well as lacustrine sediments in this region make them an attractive substitute for the existing thinner surface layers. Due to coarser textures, lower available water and weak structure, the glacial tills are better suited for placement in the lower rooting zone. Since the tills are alkaline, they may be beneficial in mixture with other more acidic materials such as acid B horizon material.

The consolidated brown shale and the sandstone in the crushed condition are physically unsuited in the upper plant root zone; the major limitations being poor structure, coarse texture, and low water-holding capacity. Both materials had an adverse effect on plant emergence when used as a seedbed medium. The brown shale contains adequate amounts of most plant nutrients and no apparent toxic elements; therefore, upon further weathering would not be detrimental to plant growth in mixture with other materials such as glacial till or loess. This sandstone, however, in addition to having undesirable physical properties, contains high manganese, sulfur and soluble salts which reduces its potential for plant growth.

The gray and black fissile shales which sometimes occur in close proximity to the coal strata are totally unsuitable for plant growth. They exhibit unfavorable physical characteristics and have chemical properties such as low pH, high potential acidity, and excessive amounts of exchangeable aluminum,

boron, sulfur and soluble salts that directly or indirectly are toxic to most plants. This is evidenced by low plant emergence, poor growth, high mortality, and excessive tissue accumulations of toxic elements in surviving plants grown on these materials. These toxic shales should be covered quickly and be placed deep enough to avoid contact with plant roots and to reduce subsequent oxidation and leaching into ground waters.

The addition of sewage sludge resulted in vastly improved growth of wheat and to a lesser extent alfalfa on all overburden materials except black fissile and gray shales. Fertilization with N-P-K was less effective than sludge, but did produce increased plant growth with major beneficial responses occurring on the unconsolidated overburden materials. It is evident that amendment of overburden materials in the upper plant root zone with major nutrient elements and organic matter from waste materials such as sludge would be a benefical practice.

Regression analysis of the results suggested properties that should be examined in evaluating overburden materials, but did not yield a useful predictive equation.

RECOMMENDATIONS

Further research is needed to field test the physical and chemical criteria developed in this work. Also, additional research is needed to more fully characterize variability encountered in the major overburden strata and to determine whether important properties are similar throughout the Illinois coal basin. In the research reported here, all materials tested were used as the entire root zone. Some of the materials appear to have properties suitable for subsoil, but not for surface use, therefore, experiments using A horizon surface material and various subsoil materials are needed. Such experiments should include mixing the B horizon with other unconsolidated materials and the addition of organic residues to the subsoil.

Overburden properties are most easily used in an elimination scheme since there are several which signal poor growth conditions even when used singly. On the other hand, no single property assures good growth conditions in any part of its range. The following properties, in the range shown, indicate a high probability of poor growth unless the condition is corrected.

Sulfur content > 1% total S
Potential acidity > 15 meq/100g
Exchangeable Al > .40 meq/100g
Extractable Mn > 60 µg/g
pH in 1:1 H_2O < 4.5
Extractable B > 1.0 µg/g
Electrical conductivity > 1.0 mmho/cm
Organic matter < 0.5% if used as top soil
Available H_2O storage < 15% by volume
Bulk density > 1.4 g/cc after packing

These are based on results of experiments in this study plus some background knowledge and literature reports. All of the materials in this study with significant S contents developed a low pH and some of the problems usually associated with it. Potential acidity is another means of detecting the same problem and requires less elaborate equipment for analysis. Low pH is usually considered an indicator of poor growth conditions, but this is primarily because it increases the availability of toxic metal cations such as Al^{+3} and Mn^{+2}. Water soluble B and high electrical conductivity are frequently associated together and with the presence of freshly exposed unweathered materials. Either can be plant inhibiting, but tend to leach from the media within a few years in humid climates such as the eastern United States. Organic matter improves cation exchange capacity, but is probably most important in providing favorable physical conditions in the surface material, e.g., reducing crusting, stabilizing structure and increasing available water

storage. Texture extremes, in this case high clay contents or high sand contents, tends to create poor physical conditions such as low available water storage and high resistance to root penetration. Bulk density of greater than 1.4 g/cc would be considered acceptable in sandy materials, but sandy materials have low water storage and low cation exchange capacity. If the properties of an overburden material are not in the unfavorable ranges listed above, then there is a high probability of good plant growth and the addition of routine amendments such as lime and fertilizer will create a good growth medium.

LITERATURE REVIEW

University scientists have conducted research on surface-mined coal lands in Indiana periodically over 28 years. Investigations have focused on cast-overburden properties (Kohnke, 1950; Byrnes and Miller, 1973); spoil bank classification and revegetation (Stiver, 1949); tree establishment, growth and development with emphasis on central hardwood species (Arnott, 1950; Tarbox, 1954; DenUyl, 1955, 1956 and 1962), natural revegetation (Byrnes and Miller, 1973), wildlife relationships (Mumford and Bramble, 1973), and socioeconomic impacts (Callahan and Callahan, 1971).

Significant research contributions also were made by other agencies and institutions on surface-mined coal lands in Indiana. Notable among these were investigations conducted by the U.S. Forest Service throughout the Central States region beginning in the mid 1940's. Forest Service research, summarized by Limstrom (1960), covered many aspects of coal-mined land including physical and chemical properties of resulting spoil, kinds of parent material, effects of grading, erosion potential, and natural and artificial reforestation. Personnel of the Indiana University Water Resources Research Center investigated effects of surface mining on ground water storage and on water quantity and quality in nearby streams (Corbett, 1965 and 1968; Agnew and Corbett, 1973).

Increased emphasis on coal production, changing mining technology, and environmental awareness beginning in the 1960's have resulted in a revitalization of interest and research on land disturbed by mining. Published literature on characteristics and reclamation of surface-mined coal lands in the Eastern Coal Region is voluminous. Funk (1962) listed 172 references on this subject covering the period 1918 to 1960. Czapowskyj (1976) prepared an annotated bibliography on disturbed land that systematically indexed 591 references by mining area, type of material and subject including geology, spoil placement and spoil characteristics. The most recent bibliography, containing more than 1300 references, on surface mining and reclamation in the Eastern Coal Province was compiled by the Argonne National Laboratory (Weiss et al., 1977). Citations in this bibliography are organized into 21 categories, including overburdens and minesoils, and stored in a data base system, BIRS, for subsequent retrieval. Symposia held at the Pennsylvania State University in 1969 (Hutnik and Davis, 1973) and at Wooster, Ohio, in 1976 (Schaller and Sutton, 1978) thoroughly explored the state-of-knowledge on ecology and reclamation of drastically disturbed lands. Current updates in the Eastern Region also have been provided by the Research and Applied Technology Symposiums on Mined-Land Reclamation sponsored by the National Coal Association in Pittsburgh, Pennsylvania, in 1973 and Louisville, Kentucky, in 1974 through 1977 (National Coal Assn., 1973-77).

Information provided by many of these investigations has been useful in

the formulation of state laws (State of Indiana, 1967) regulating surface mining and reclamation, and subsequent development of guidelines and standards (State of Indiana, 1973) for their administration. However, with the enactment of federal legislation in 1977 (United States of America, 1977), additional and more specific knowledge is needed on mine overburden properties, placement and plant growth potential.

Numerous investigations have been conducted in recent years on properties of "cast overburden" resulting from modern mining methods (Berg and Vogel, 1968 and 1973; Cummins et al. 1965; Czapowskyj, 1973; Massey, 1972; Massey and Barnhisel, 1972; Plass, 1969; Plass and Capp, 1974; Plass and Vogel , 1973; VanLear, 1971; Yamamoto, 1975). A current example is the work of Barnhisel (1977), who investigated spoil (cast overburden) properties and forage yields on four types of existing surface mine coal spoils, from neutral to very acid, in western Kentucky. Barnhisel found that phosphorus and water are more commonly the limiting factors in obtaining an adequate vegetation cover than the acidic nature of spoils. Further, he indicated that acidic spoils tend to be much more droughty than adjacent non-acid spoils as the result of a restricted rooting depth. Armiger et al. (1975) noted that strip-mine spoils vary greatly in chemical composition and often require markedly different reclamation and revegetation procedures to alleviate toxic conditions. They stressed the need for chemical analysis of spoil, in association with greenhouse plant growth studies, to establish the presence of toxic elements and to select amendments for effective planning of field revegetation. Such information on "cast overburden" properties is useful for classifying existing conditions for plant growth, recommending amendments, and developing guidelines, but generally is "after-the-fact" in mining and grading operations.

Although the general literature on minesoils and plant growth is too expensive and varied for inclusion, several references pertinent to the topic of overburden properties before mining and their relation to plant growth potential are appropriate. Limstrom and Mertz (1951) described the stratigraphy of overburden highwalls in several coal-mining districts in Ohio in the late 1940's. They extracted samples from each highwall stratum and from cast-overburden surfaces for laboratory analysis. Analysis was limited to identification of type and thickness of soil and geologic strata, particle size distribution, pH, and available phosphorus and potassium. Data were presented in tabular form with some description of resultant spoil, but with little interpretation for plant growth. May and Berg (1967) determined acidity of highwall strata of five major coal seams in eastern Kentucky. They reflected that geological and chemical characteristics of the overburden will aid greatly in identifying and properly handling acid-bearing materials during the mining operation. In subsequent investigations, Berg and May (1969) measured acidity and plant-available phosphorus in overburden strata of six Kentucky mines. Extremely acid strata were observed in all mines and plant-available phosphorus generally was very low.

Krause (1973) indicated that "the key to achieving a predictable growing medium lies in identifying which combination of overburden strata is best suited for the survival and growth of vegetation", which is the primary objective of this project. Krause presented information on highwall studies in Ohio which included geology and thickness of strata and analyses for pH,

total soluble salts, and 14 compounds and elements. He showed by diagrams how mining operations could be handled to provide improved growing media. A similar study conducted in eastern Kentucky (Despard, 1974) concerned the identification of acid strata in the overburden and avoidance of its placement in the plant growth zone of the cast-overburden. Despard emphasized the importance of overburden analysis before mining is begun and indicated that color, pyrite content, and pH of exposed overburden could serve as field guides to determine potential toxicity. In a publication on rehabilitation potentials and limitations of surface-mined land in the northern Great Plains, Packer (1974) listed analysis of chemical and physical characteristics of surface-mine overburden before mining as the first priority in research needs.

Based on extensive overburden and minesoil research in the Appalachian Coal Region, Grube et al. (1974) indicate that when specific overburden properties are known in advance of mining it is possible to plan the operation and subsequent reclamation to take advantage of favorable physical and chemical properties and avoid possible toxicities. They stress that advance information should include acid-base accounts, concentrations of plant nutrients, and physical stability of all overburden horizons when exposed to the weather. The acid-base account was further emphasized as a primary property of overburden materials by Sobek et al. (1976). Acid-toxic overburden is defined by the measurement and interpretation of paste pH, total or pyritic sulphur, and neutralization potential. Sobek et al. state that the test for acid-toxic materials is a paste pH reading below 4.0 or an acid-base account that shows a net deficiency of more than 4.5 MT calcium carbonate equivalent per 907 MT of material. When the maximum acid calculation is more than 4.5 MT calcium carbonate equivalent per 907 MT of material in excess of the neutralization potential the material is considered toxic regardless of its pH.

Schroer (1978) recognized that at present there is little information on the chemical and physical properties of overburdens from depths greater than 1.5 meters. Therefore, he sampled and analyzed 20 soil series to a depth of 1.5 meters and several hundred underlying overburdens to a depth of currently mined or mineable coal in the lignite region of North Dakota. Soil horizons were analyzed by standard soil characterization methods. Schroer observed that physical and chemical properties varied widely with depth and across the surface within and between mine areas. These variations were associated with the stratified nature of the geologic materials that constitute the overburdens. Schroer concluded that on site sampling will be needed at future mine sites to assess overburden properties significant to reclamation.

Since the lower strata of mineland materials differ drastically from overlying soils, special techniques may be required for their analysis. This is particularly true in the western states where salinity associated with nutrient deficiencies is common in these materials. Therefore, Sandoval and Power (1977) developed suggested guidelines for sampling and chemical analysis of mine spoils and overburden samples to meet the need for uniform laboratory methods to evaluate plant growth capabilities and limitations associated with coal lands in western United States. Appropriate uniform procedures also are needed in the Eastern Coal Region for collection, preparation and analysis of the physical and chemical properties of consolidated overburden strata.

PROCEDURES

MATERIAL COLLECTION

Eighteen surface mine overburden materials for analysis and growth trials were selected from five sites in southwestern Indiana (Figure 1). Sites were chosen that spanned the range of major overburden types in the eastern edge of the Illinois Coal Basin; then strata were selected at these sites that appeared physically adapted to root zone construction, and commonly occurred over coal seams mined in the region. Thus, materials excluded were massive, hard rock materials such as limestone and layers too thin to be handled separately by draglines or shovels, except that some surface soil materials (A horizons) were collected for comparison. The materials collected are representative of the major strata important in Indiana mineland reclamation. Twelve are unconsolidated materials, including A and B horizons, loess, lacustrine deposits, and glacial tills, and six are rock strata that break and weather easily.

Table 1 lists the materials sampled and their location by county, mine, and rock formation; soils are identified by series and horizon. Logs representative of the highwalls at three mine sites are shown in Tables 2, 3, and 4. Several hundred kilograms of such material were collected from recently exposed highwalls or bench cuts in November-December, 1976. Additional samples of ten strata materials were collected again in July and December 1977 at different locations within the same mine to obtain some measure of field variability.

CHEMICAL AND PHYSICAL ANALYSES

The rock materials, shales and sandstone, were crushed in a "chipmunk jaw" crusher until all particles were less than 2 cm in diameter. Crushed samples were used in the greenhouse and laboratory studies. Uncrushed material, partially broken by digging from the highwall and handling, was used in the outdoor plant growth studies in large containers. Crushing was necessary for some of the studies and approximates conditions expected after blasting, casting, grading, and short term exposure. Each overburden material was air dried, thoroughly mixed, and subsampled for analysis and growth trials. Materials used in chemical and physical analyses were crushed to pass a 2 mm sieve to conform to standard soil analysis procedures.

The chemical character of the material was determined by elemental analysis for plant-available P, exchangeable K, Ca, Mg, Na, Al, extractable Mn, B, and total sulfur. Other chemical analyses included pH, buffer pH, potential acidity, cation exchange capacity, organic matter, and electrical conductivity. Physical properties analyzed were particle size distribution, moisture retention and bulk density. The analyses are listed in Table 5 along with

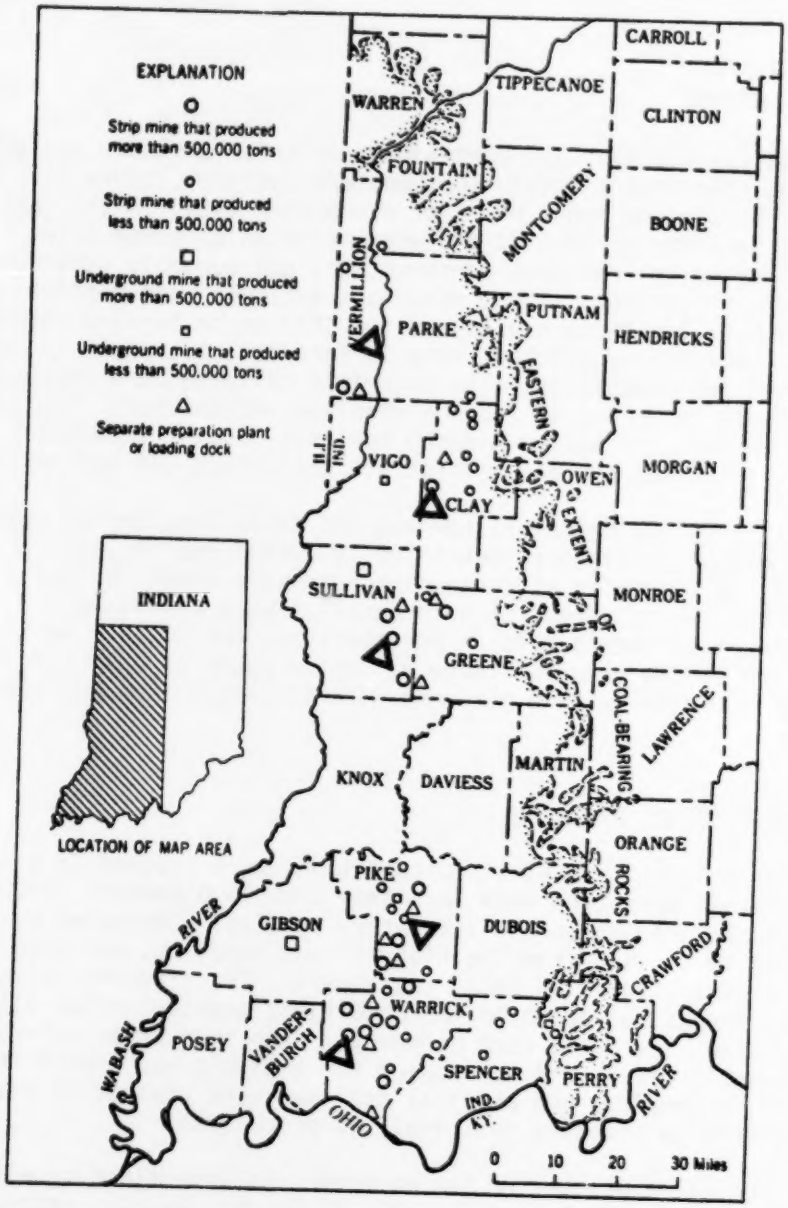

Figure 1. Map of southwestern Indiana showing active coal mines and selected study sites (△). Modified from Hutchison, 1971.

TABLE 1. OVERBURDEN MATERIALS SAMPLED.

Overburden material	Location		Rock Formation
	Mine	County	
Hosmer A-Horizon	Ayrshire	Warrick	
Iva A-Horizon	Chinook	Clay	
Zanesville A-Horizon	Ayrcoe	Pike	
Hosmer B-Horizon	Ayrshire	Warrick	
Iva B-Horizon	Chinook	Clay	
Zanesville B-Horizon	Ayrcoe	Pike	
Unweathered Illinoian Till	Chinook	Clay	
Weathered Illinoian Till	Chinook	Clay	
Wisconsin Till	Universal	Vermillion	
Kansan Till	Universal	Vermillion	
Loess (weathered)	Ayrshire	Warrick	
Lacustrine Sediments	Ayrshire	Warrick	
Black Fissile Shale 1	Ayrcoe	Pike	Dugger
Black Fissile Shale 2	Ayrshire	Warrick	Dugger
Gray Marine Shale	Ayrshire	Warrick	Dugger
Gray Fluvial Shale	Minnehaha	Sullivan	Dugger
Brown Weathered Shale	Ayrshire	Warrick	Dugger
Sandstone	Ayrcoe	Pike	Dugger

TABLE 2. GENERALIZED HIGHWALL LOG FOR AMAX AYRCOE MINE IN PIKE COUNTY. SOIL TYPE: ZANESVILLE SILT LOAM.

Type of Material	Average thickness
	meters
Soil Solum (A and B horizon)*	1.3
Loess	4.0
Sandstone*	0.8
Gray Shale	6.5
Coal V B	0.2
Sandstone	0.5
Gray Shale	5.0
Coal V B	0.2
Gray Shale	5.0
Asiderite plus Gray Shale	0.2
Black Fissile Shale	0.1
Alum Cave Limestone	0.3
Black Fissile Shale*	1.0
Springfield Coal V	1.3

*Overburden materials sampled.

TABLE 3. GENERALIZED HIGHWALL LOG OF AMAX CHINOOK MINE IN CLAY COUNTY FROM DRILL LOG. SOIL TYPE: IVA SILT LOAM.

Type of Material	Average thickness
	meters
Soil Solum (A and B horizon)*	1.5
Weathered Illinoian Till*	1.5
Unweathered Illinoian Till*†	10.5
Gray Shale	0.3
Silty Sandstone	0.6
Gray Shale	2.4
Mixed Silty Sandstone and Shale	4.8
Dark Gray Shale	1.0
Coal III	1.5
Underclay	0.5
Black Fissile Shale	1.5
Coal III	0.5

*Overburden material sampled.

†Weathered till was differentiated from unweathered due to its brown color and absence of calcium carbonates.

TABLE 4. GENERALIZED HIGHWALL LOG FOR AMAX AYRSHIRE MINE IN WARRICK COUNTY. SOIL TYPE: HOSMER SILT LOAM.

Type of Material	Average thickness
	meters
Soil Solum (A and B horizon)*	1.5
Loess*	6.0
Brown Shale*	4.5
Gray Shale*	9.0
Black Fissile Shale*	0.8
Upper Millersburg Coal	1.0
Layered Dark Gray Limestone and Dark Gray Shale Parting	1.0
Lower Millersburg Coal	1.0

*Overburden materials sampled.

the extractant used for elements and reference to the procedure. A brief summary of procedures used is given in Appendix A.

PLANT GROWTH STUDIES

Two greenhouse projects and one large outdoor container project evaluated the growth of plants with and without amendments on these overburden materials.

Greenhouse project one, conducted in the spring of 1977, was a randomized incomplete block design with 10 overburden materials, 3 plant species, 2 fertilizer treatments and 2 replications. Overburden materials were Iva A and B horizon, weathered and unweathered Illinoian till, Wisconsin till, loess, black fissile shale No. 1, gray marine shale, brown shale and sandstone. Plant species included alfalfa (Medicago sativa), wheat (Triticum vulgare), and eastern white pine (Pinus strobus). The fertilizer treatments consisted of a control and 10-10-10 applied at a rate equivalent to 74 kg/ha each of N, P_2O_5, and K_2O mixed throughout the overburden sample. Approximately 5.5 kg of each material were placed in black, four liter clay pots. Twenty alfalfa or wheat seeds were planted or four eastern white pine seedlings about 5 cm tall were transplanted in each pot. After 12 weeks the plants were harvested, dried at 65 C, and weighed.

Greenhouse project two was conducted in 1978 to further evaluate the 10 overburden materials used in the first study plus 5 additional materials, Hosmer and Zanesville A and B horizons and lacustrine sediment. The experiment consisted of three randomized complete blocks, each containing a factorial treatment design of the 15 materials; two species, wheat and alfalfa and three amendment treatments, sewage, fertilizer plus lime, and control. The sewage sludge treatment was 5.5 metric tons per hectare (mt/ha dry wt basis, calculated assuming 2240 metric tons of soil in the plow layer of one hectare). The sludge had 2% solids. The fertilizer and lime treatments were tailored to each material. Lime, if needed, was added to adjust pH to 6.5, and N, P, K was added according to Purdue Soil Test Laboratory's recommendations for the species based on soil test results. One material, the black fissile shale, received 22 mt/ha lime. More was needed to bring the pH up to 6.5, but 22 mt/ha was considered to be the maximum practical application based on field experience. About 2 kg of overburden material with sludge or fertilizer incorporated was used in each 2 liter clay pot. Twenty wheat or alfalfa seeds were planted per pot. The alfalfa was inoculated with rhizobium. The percent and rate of emergence, periodic height growth, and final dry weight were recorded.

The large outdoor container study was established in the spring of 1978, and will be continued through 1979. Approximately 100 liters (26 gallons) of overburden material were placed in plastic lined cans fitted with drains for leachate collection. Thirty containers were arranged in three randomized blocks, each containing ten materials. The materials used were the A and B horizon of the Iva soil, black fissile shale No. 1, sandstone, gray marine shale, brown shale, weathered Illinoian till, unweathered Illinoian till, Wisconsin till and loess. Each container was initially planted to oats (Avena sativa) at a rate of 5 g per container in the early spring. After

TABLE 5. PROCEDURES USED IN CHEMICAL AND PHYSICAL CHARACTERIZATION OF THE OVERBURDEN.

Characteristics	Procedure	Reference
Elemental:		
available P	Bray P_1 extraction	(Knudsen, 1975)
exchangeable K	1 N NH_4Ac extraction	(Carson, 1975)
exchangeable Ca	1 N NH_4Ac extraction	(Walsh, 1971)
exchangeable Mg	1 N NH_4Ac extraction	(Walsh, 1971)
exchangeable Na	1 N NH_4Ac extraction	(Walsh, 1971)
exchangeable Al	1 N KCl extraction	(Black, 1965)
extractable Mn	0.1 N H_3PO_4 extraction	(Whitney, 1975)
extractable B	Hot water extraction	(Whitney, 1975)
extractable Mo	pH 3.3 $NH_4C_2O_4$ extraction	(Black, 1965)
total S	LECO induction furnace	(Walsh, 1971)
Other chemical:		
pH	1:1 soil to water	(McLean, 1975)
buffer pH	SMP buffer pH	(McLean, 1975)
cation exchange capacity	sum of extractable acidity and exchangeable K, Ca, Mg, Na	(Black, 1965)
organic matter	Walkley-Black wet oxidation	(Black, 1965)
soluble salts	electrical conductivity	(Black, 1965)
potential acidity	H_2O_2 oxidation and titration with NaOH	(Smith et al., 1973)
Physical:		
particle size	pipette method	(Black, 1965)
moisture retention	pressure membrane and pressure plate	(Black, 1965)
bulk density	repacked samples	(Black, 1965)

four weeks, the oats were harvested, dried, and weighed. The containers then were divided into split plots for planting of two one-year seedlings each of Virginia pine (Pinus virginiana) and yellow-poplar (Liriodendron tulipifera). Tree height and root collar diameter were measured immediately after planting and at the end of the first growing season. Leachate from the large containers was collected periodically and analyzed for pH, soluble salts, Ca, P, and K to determine the extent of nutrient loss in early stages of weathering.

STATISTICAL PROCEDURES

Data on plant growth were examined by analysis of variance. Significant treatment combinations were ordered and differences between observed levels (means) were compared with a Newman-Kuels test or Duncan's Multiple Range test. The relationships among physical and chemical characteristics were examined through correlation analysis. Physical and chemical properties of overburden materials were then added in a stepwise multiple regression program to select 1) the property most highly correlated with plant growth, and 2) those properties significantly related to plant growth in multiple correlations.

RESULTS AND DISCUSSION

PHYSICAL AND CHEMICAL OVERBURDEN PROPERTIES

Physical and chemical properties of selected overburden materials were analyzed to determine their relationship to plant growth characteristics. These properties were determined for disturbed overburden samples and are representative of materials used in greenhouse pot culture and outdoor container studies. The data do not represent properties of various overburden materials in the natural undisturbed state. It is probable, however, that they closely reflect the ultimate cast overburden in which various strata, both consolidated and unconsolidated, have been subjected to disturbance by blasting, excavation, mixing, replacement, grading and short-term weathering.

Use of disturbed materials is appropriate and generally consistent with standard soil test procedures for determination of chemical properties and particle size distribution, except for artificial crushing of rock strata. Physical properties such as bulk density and moisture retention, especially at 1/3 bar tension, are normally determined on undisturbed samples indicative of field conditions. In this study, however, bulk density and moisture retention measurements are based on disturbed and repacked materials and therefore deviate from anticipated field values for the soil solum and other unconsolidated material, but is the only feasible approach for rock strata. Although some of these procedures deviate from standard soil analysis methods, they attempt to approximate the restructured conditions typical of reclaimed mineland.

Physical Properties

Bulk density, particle size distribution and moisture content at 1/3 bar and 15 bar tensions were determined on three replicates each of 18 overburden materials (Table 6). In respect to soil-plant growth factors, these properties are important in water relations, aeration, ion exchange and nutrient element availability. Available water-holding capacity was calculated as the difference in water retention at 1/3 bar tension and 15 bar tension. The textural class for each material is based on the proportion of sand, silt and clay size particles. Three soil series, Hosmer, Iva and Zanesville, which are representative of upland soils in the mining region were included for analysis of A and B horizon soil material.

Based on the range of values for the physical properties measured, the 18 overburden materials separated into essentially three groups (Table 6). These are:

1. A and B horizon soil, loess and lacustrine sediment. These are predominately silt loams containing 84 to 99% silt plus clay size

TABLE 6. PHYSICAL PROPERTIES OF SELECTED OVERBURDEN MATERIALS.

Overburden material	Bulk density	Moisture retention			Particle size			Text. class
		1/3 bar	15 bar	Avail.	Sand	Silt	Clay	
	g/cc	------ % by vol. ------			------- % ------			
A Horizon								
Hosmer	1.09	24.5	7.6	16.9	4.3	81.3	14.4	sil
Iva	1.21	25.8	6.8	19.0	16.2	72.7	11.1	sil
Zanesville	0.98	27.1	6.5	20.6	4.7	87.2	8.1	si
B Horizon								
Hosmer	1.12	31.1	11.3	19.8	1.6	74.1	24.3	sil
Iva	1.09	29.7	13.5	16.2	9.5	61.1	29.4	sicl
Zanesville	1.13	32.5	9.9	22.6	2.3	74.1	23.6	sil
Loess	1.18	30.2	10.0	20.2	10.9	70.8	18.3	sil
Lacustrine	1.12	46.0	20.8	25.2	1.0	74.1	24.9	sil
Glacial Till								
Wisconsin	1.52	19.0	8.0	11.0	48.2	37.0	14.8	l
Unw. Illinoian	1.51	18.3	7.1	11.2	53.9	31.8	14.3	gsl
Wx. Illinoian	1.26	25.4	12.0	13.4	30.0	49.1	20.9	l
Kansan	1.60	18.2	8.3	9.9	51.3	33.4	15.3	l
Black Fissile Shale								
#1	1.41	20.4	11.1	9.3	65.1	31.5	3.4	gsl
#2	1.31	19.4	14.3	5.1	68.5	28.1	3.4	gsl
Gray Shale								
Marine	1.56	26.6	13.1	13.5	8.4	70.6	21.0	gsil
Fluvial	1.34	18.2	12.4	5.8	17.3	67.3	15.4	gsil
Brown Shale	1.49	25.5	13.1	12.4	22.7	58.9	18.4	gsil
Sandstone	1.46	17.1	4.7	12.4	68.4	27.8	3.8	gsl

particles. The bulk density was low, ranging from 0.98 to 1.21 g/cc, for all materials. These materials also exhibited the greatest moisture retention with available water capacity in the range of 16 to 25% by volume. The lacustrine sediment with 99% silt plus clay content and nearly 2.0% organic matter had the highest, 25%, available water.

2. Wisconsin, unweathered and weathered Illinoian and Kansan glacial tills. The tills are mostly loam textures with silt plus clay contents of 46 to 52% and moderate bulk densities of 1.26 to 1.60 g/cc. The bulk densities are considerably lower than the tills normally exhibit in their undisturbed state. Available water-holding capacity ranged from 10 to 13% which averages slightly more than one-half the water-holding capacity of the soil solum, loess and lacustrine materials.

3. Black fissile shale, gray shale, brown shale and sandstone rock materials that were ground to particle sizes less than 2 cm diameter. The resultant texture of these materials, likely due to inherent rock characteristics, varied from silt loam with 77 to 92% silt plus clay content for the gray and brown shales to sandy loam with 32 to 35% silt plus clay for the black fissile shale and sandstone. All ground rock materials contained more than 15% coarse fragments greater than 2 mm; thus, the textural classes were designated gravelly. Bulk densities for the fractionated rock materials ranged from 1.31 to 1.56 g/cc and available water capacity from 5 to 13% by volume. The lowest water retention, 5 to 9% available, occurred in the black fissile shale and gray fluvial shale materials. The high gravel and/or sand content of these materials likely influenced water retention capacity.

Available moisture in the rooting medium is considered a major environmental factor in mineland revegetation. The quantity of available water is directly influenced by soil texture with silt loams, loams and clay loams generally exhibiting the greatest water-holding capacity (Brady, 1974). From this standpoint, it is evident that the soil solum, loess, lacustrine sediment and glacial tills analyzed in this study offer the best potential for plant-available water. The higher gravel and sand content of most rock materials reduce water retention capacity and tend to be more droughty. The occurrence of high soluble salts, such as levels present in the fissile shales, also may influence available water.

Under field conditions, the depth of soil material of a given textural class as well as layering of materials of different textures will affect water distribution and availability to plants. Also, materials with higher organic matter content will indirectly influence water retention through improvements in soil structure. Therefore, in the reconstruction of mineland soils, it is important to consider texture and other physical properties of soil materials regarding their final placement and depth in the cast overburden.

Chemical Properties

Elemental content of the eighteen overburden materials is presented in Table 7. Available phosphorus, extractable with weak acid, is generally very low in all materials with the exception of the two black shales. The

TABLE 7. MEAN ELEMENTAL CONTENT OF SELECTED OVERBURDEN MATERIALS.

Overburden material	Available		Exchangeable				Extractable		Total
	P	K	Ca	Mg	Na	AT	Mn	B	S
	kg/ha		- - - - - -meq/100g- - - - - -				µg/g		%
A Horizon									
Hosmer	6	76	3.7	1.2	0.03	.01	11	0.25	0.09
Iva	18	107	8.0	1.2	0.02	.00	17	0.30	0.12
Zanesville	6	132	2.4	1.0	0.01	.06	35	0.25	0.11
B Horizon									
Hosmer	2	85	2.7	2.7	0.09	.26	6	0.25	0.16
Iva	6	222	9.4	6.6	0.14	.04	28	0.35	0.10
Zanesville	6	60	3.4	3.5	0.08	.15	10	0.25	0.16
Loess	12	98	4.6	4.4	0.36	.02	18	0.25	0.11
Lacustrine	3	143	18.3	4.7	0.15	.01	19	0.40	----
Glacial Till									
Wisconsin	2	74	16.4	1.9	0.03	.00	9	0.25	0.14
Unw. Illinoian	3	106	15.3	2.0	0.08	.01	21	0.30	0.27
Wx. Illinoian	14	113	7.3	5.3	0.17	.01	51	0.25	0.11
Kansan	1	69	18.1	2.4	0.00	.00	11	1.00	----
Black Fissile Shale #1	61	146	30.3	7.8	0.19	.64	67	1.30	3.27
#2	134	13	13.3	7.7	0.02	.43	72	2.95	3.63
Gray Shale									
Marine	6	435	10.7	5.2	2.21	.02	44	3.05	0.65
Fluvial	5	206	4.9	3.4	0.16	.02	4	2.50	0.56
Brown Shale	3	224	7.4	7.8	0.91	.01	28	0.40	0.13
Sandstone	8	90	9.7	5.0	0.22	.00	62	0.45	0.62

relatively high organic matter content in the black shales (Table 8) probably accounts for their P reserves. Abailable potassium was also in short supply in most materials. The Iva B horizon and the lacustrine material K levels are considered "medium" to "high" by Indiana soil test standards. The gray shales were high in K and one of the black shales had a "medium" test level.

Exchangeable calcium and magnesium levels in all of the materials appear to be adequate. Where Mg is high and equals or exceeds calcium, in the brown shale, for example, there is a tendency toward poor structural stability.

Sodium levels are not high enough to be of concern in any of the materials. The highest levels are in the marine shale where exchangeable Na occupies about 4% of the exchange capacity.

Exchangeable Al is very high in the two black shales which are also very acidic. Aluminum toxicity is certainly likely to occur on those materials and may be a problem in the Hosmer B where there is a pH of 4.7 and a significant quantity of exchangeable Al, 0.26 meq/100g. The Al levels are generally lower than those of West Virginia spoils reported by Plass and Vogel (1973).

Manganese is adequate in all of the materials. Manganese toxicity most commonly occurs in soils containing high levels of Mn that are also acidic and poorly drained such that reduction of Mn occurs. The levels in the black shales and sandstone might be a problem for sensitive crops grown under poorly aerated conditions.

Hot-water-extractable boron levels are high enough for most plants in all eighteen materials. The levels near 3 ppm in the gray and black shales are potentially toxic to sensitive plants (Sauchelli, 1969; Rogers, 1947).

There is probably adequate S for normal plant growth in all of these materials. However, the levels in the black and gray shales and sandstone account for their high potential acidity (Table 8).

The acid-base status of these overburden materials is reflected in the pH, buffer pH, potential acidity, cation exchange capacity (CEC) and percentage base saturation reported in Table 8. The lacustrine material, the brown shale, and the glacial tills, with the exception of the weathered Illinoian till, contain significant amounts of carbonates, are alkaline, and near 100% base saturation. The A and B horizons and the loess are acid, but within a tolerable range for many plants and could be brought to levels suitable for most crops with reasonable lime additions. The black and gray shales and sandstone, however, contain significant amounts of S and high potential acidity (Table 8) which would require very high amounts of lime to correct. In spoil bank classification for tree growth, Limstrom (1960) rated materials with pH below 4.0 as marginal to toxic.

The electrical conductivity (E.C.) of the soil solution is high enough to be detrimental to plant growth only in the black and gray shales and sandstone. Sensitive plants are retarded by water exceeding 1 mmho/cm in conductivity (Nieman and Shannon, 1976). Cummins et al (1965) reported that mineland spoils

TABLE 8. MEAN CHEMICAL PROPERTIES OF SELECTED OVERBURDEN MATERIALS.

Overburden material	pH	Buffer pH	Potent. acidity	CEC	Base Sat.	E.C.	Organic matter	$CaCO_3$ Equiv.
			meq/100g	meq/100g	%	mmhos/cm	%	%
A Horizon								
Hosmer	5.3	6.8	N/A†	11.5	44.9	0.10	1.22	N/A
Iva	6.9	N/A*	N/A	12.0	77.9	0.19	1.11	N/A
Zanesville	4.9	6.5	N/A	12.6	29.4	0.16	2.28	N/A
B Horizon								
Hosmer	4.7	6.2	N/A	16.9	33.1	0.09	0.54	N/A
Iva	5.5	6.8	N/A	23.7	69.5	0.20	0.40	N/A
Zanesville	5.0	6.4	N/A	17.0	42.0	0.08	0.25	N/A
Loess	5.8	6.8	N/A	14.8	65.6	0.07	0.24	N/A
Lacustrine	8.0	N/A	N/A	23.3	100.0	0.40	1.96	15.3
Glacial Till								
Wisconsin	8.2	N/A	N/A	18.3	100.0	0.17	0.36	24.5
Unw. Illinoian	7.9	N/A	N/A	17.7	99.0	0.81	0.59	25.3
Wx. Illinoian	6.8	N/A	N/A	16.3	79.6	0.18	0.26	.1
Kansan	7.7	N/A	N/A	20.6	100.0	0.80	0.51	15.2
Black Fissile Shale								
#1	3.7	4.6	130.5	67.4	69.1	5.47	4.17	N/A
#2	2.7	3.2	465.2	62.4	33.6	8.40	7.35	N/A
Gray Shale								
Marine	6.6	N/A	16.5	22.1	83.3	2.12	2.18	N/A
Fluvial	5.4	6.7	69.1	12.7	68.1	1.40	8.62	N/A
Brown Shale	7.3	N/A	N/A	18.2	90.0	0.21	.41	3.6
Sandstone	5.5	7.2	18.0	16.6	91.0	2.09	1.22	N/A

*Not applicable, determined only on samples of pH 6.5 or less.
†Not applicable, determined only on samples with S > 0.5%.

with electrical conductivity of 2 mmhos/cm was marginal and 3 mmhos/cm was toxic for plant growth.

In summary, the chemical properties of the overburden materials suggest several problems likely to occur in using the materials for plant growth media. Acid formation from the S in the black and gray shales and the sandstones is likely to cause many problems in addition to their high conductivity and B contents. The low P and K levels in many of the materials could be overcome with fertilizer and lime could be used to raise the pH and reduce exchangeable Al on the more acid unconsolidated materials. Toxicity from B or Mn appears likely only on the same materials that have significant S content and associated acidity.

The coefficients of variation for the chemical properties presented in Table 9 indicate the difficulty in characterizing overburden materials with limited sampling. In general, the rock materials exhibited the greatest variability in chemical properties. Some of this is probably a result of segregation that occurs in crushing and screening in addition to the natural variation in the field. The relatively great variation in important properties such as available P and K, electrical conductivity, and organic matter content explain inconsistent and unusual results frequently encountered in both field and greenhouse growth experiments.

Correlation of Overburden Properties

The results of a simple correlation analysis of twelve physical and chemical properties are presented in Table 10. The variability of properties within any one material (Table 9) precludes the possibility of finding very high correlations. However, some correlations that are common in soil materials did appear, e.g., the positive correlation (.68) between pH and percentage base saturation (B.S.), and the relationship of organic matter (O.M.) and sulfur (S). The shales tended to be high in organic matter, sulfur, manganese (Mn), and electrical conductivity (E.C.), and quite different from most of the other materials in these properties. Thus, those three properties appear correlated, but if the unconsolidated materials were examined separately, there would be little relationship among them. Bulk density (B.D.) is negatively correlated with percent silt, and this is expected in these repacked samples where high bulk density is associated with higher percentage coarse material. The negative relationship between electrical conductivity and percent silt is primarily due to the two black shales which contained high soluble salts and were low in percent silt in this analysis. The relationship is certainly not expected to be a valid generality for overburden materials. The strong positive correlation between boron (B) and potassium (K) is interesting and may be useful in initial screening for potential boron problems based on potassium tests which are done much more routinely.

PLANT GROWTH STUDIES-GREENHOUSE

Wheat and Alfalfa Yields

The first greenhouse experiment utilized three plant species grown on ten

TABLE 9. RELATIVE VARIATION, C.V. (%), IN CHEMICAL PROPERTIES WITHIN STRATA BASED ON ANALYSIS OF SELECTED OVERBURDEN MATERIALS COLLECTED AT THREE SAMPLING POINTS AT THREE TIMES.

Overburden material	P	K	Ca	Mg	Na	Mn	B	pH	CEC	Base Sat.	E.C.	O.M.
	Coefficient of Variation (%)											
A Horizon-Iva	37	28	18	17	25	127	20	5	12	5	54	11
B Horizon-Iva	33	9	19	6	34	34	55	5	6	4	59	18
Loess	77	27	29	37	106	37	0	11	16	15	61	15
Wisconsin Till	25	44	4	5	22	46	0	1	4	0	27	33
Unw. Illinoian Till	33	23	21	15	45	30	10	2	19	2	19	22
Wx. Illinoian Till	29	17	6	4	13	35	0	1	6	3	31	13
Black Fissile Shale #1	129	127	27	33	132	50	28	46	69	37	72	26
Gray Marine Shale	17	36	78	44	141	45	39	15	52	6	80	50
Brown Shale	69	36	14	16	106	76	28	5	25	5	61	34
Sandstone	64	75	7	20	131	23	6	5	12	9	71	77

TABLE 10. MATRIX OF CORRELATION COEFFICIENTS(r) FOR MEASURED PROPERTIES OF SELECTED OVERBURDEN MATERIALS.

Properties	BS	OM	EC	BD	K	P	pH	Silt	Al	Mn	B	S
S	.21	.76	.78	.45	-.03	.05	-.41	-.59	-.07	.81	.18	1.00
B	.28	.29	.36	.48	.78	.12	-.18	-.32	-.18	.31	1.00	
Mn	.28	.72	.82	.50	.15	.35	-.34	-.59	-.23	1.00		
Al	-.49	-.27	-.24	-.44	.09	-.20	-.45	.29	1.00			
Silt	-.73	-.36	-.78	-.94	-.15	.25	-.18	1.00				
pH	.68	-.37	-.31	.24	.05	-.19	1.00					
P	-.08	.03	.05	-.22	-.10	1.00						
K	.33	.01	.01	.29	1.00							
BD	.77	.31	.72	1.00								
EC	.38	.75	1.00									
OM	.12	1.00										
BS	1.00											

overburden materials, both fertilized and unfertilized (control). It was a preliminary experiment, only partially replicated, designed to refine the technique, determine which species were adequate for testing and furnish preliminary results. The dry weight of plant material produced is presented in Table 11 with the exception of the white pine. In the limited duration (12 weeks) of the experiment, white pine seedlings growth was quite small, .06 to .55 g dry weight per pot, and produced no statistically significant differences among materials or treatments. Wheat and alfalfa yields in unfertilized materials indicate that Iva A horizon was the best growth medium; for wheat alone Iva A was significantly better than the black fissile shale and for alfalfa better than all materials except Iva B horizon, Wisconsin till, weathered Illinoian till and brown shale. Differences among materials became much more pronounced when they were fertilized. The fertilizer response was significant, as expected, for most materials. The lack of response in the black and gray shales was probably due to the presence of limiting factors such as low pH, high B, and high soluble salt content (Tables 7 and 8). Plants in greenhouse pot studies were watered by overhead sprinkling on a schedule designed to prevent wilting. However, the water storage capacity of various materials could become important between waterings.

The second greenhouse experiment conducted for eight weeks included 15 materials, 2 species, 3 treatments, and 3 replications (Table 12). The fertilizer and sludge treatments included lime where it was needed. Across all treatments and species, the lacustrine material produced the highest yields with the exception of the control and sludge treatments on alfalfa, and the black fissile shale produced the least. The A horizon materials all produced high yields, similar to each other, and not statistically different from the lacustrine material for alfalfa. The materials tend to form three groups if one considers the mean of treatments and species. The A horizon and lacustrine materials produce the best growth and usually are the same statistically. The B horizons, loess, glacial tills, brown shale, and sandstone tend to form a second group which are not significantly different from each other. Finally, the gray shale and black shale are clearly inferior growth media.

As in the firest greenhouse experiment, the addition of fertilizer usually improved the growth of wheat with significant responses occurring on B horizon, lacustrine, weathered Illinoian till and gray shale materials. Alfalfa yield increased with fertilization on 11 of the 15 materials, but overall, yield differences in comparison with the controls were not significant. In contrast, the sludge treatment produced wheat yields significantly greater than the controls on all materials except the black and gray shales. Growth responses to sewage sludge added may be due primarily to NH_4^+-N estimated at 200 kg/ha and P at 165 kg/ha for the sludge rate applied. Alfalfa yields also were generally higher on materials with sludge treatment than on the control, but differences in mean yield were not significant except for alfalfa grown on loess.

It appears that the only overburden tested that is equal to A horizon materials is the lacustrine material. This is not surprising when the physical and chemical properties are compared. The lacustrine material had a high pH, high exchangeable Ca and Mg, high available water-holding capacity and the

TABLE 11. EFFECT OF OVERBURDEN MATERIAL AND TREATMENT ON YIELD OF WHEAT AND ALFALFA IN THE FIRST GREENHOUSE STUDY.

Overburden material	Wheat		Alfalfa	
	Control	Fert.	Control	Fert.
	- - - - - - - - g per pot - - - - - - - -			
A-horizon, Iva	2.10a*	3.87ab	2.33a	4.48a
B-horizon, Iva	0.94ab	4.02ab	1.19ab	4.30a
Loess	0.72ab	3.27b	0.33b	2.88abc
Wisc. Till	1.01ab	2.85b	0.84ab	2.15bc
Unw. Ill. Till	2.26a	5.25a	0.22b	3.33ab
Wx. Ill. Till	1.08ab	3.14b	0.89ab	3.49ab
Blk. Fis. Shale	0.06b	0.78d	0.00b	0.10d
Gray Mar. Shale	1.35ab	1.24cd	0.05b	0.02d
Brown Shale	0.70ab	2.30bc	0.77ab	3.34ab
Sandstone	0.67ab	2.31bc	0.01b	1.47c

*Within columns, values among materials followed by same letter are not significantly different at the 5% level. Within rows, values between treatments differing by more than 1.07 g for either species are significantly different.

TABLE 12. EFFECT OF OVERBURDEN MATERIAL AND AMENDMENTS ON GROWTH OF WHEAT AND ALFALFA IN THE SECOND GREENHOUSE EXPERIMENT. EACH VALUE IS A MEAN OF THREE REPLICATES.

Overburden material	Wheat			Alfalfa		
	Control	Sludge	Fert.	Control	Sludge	Fert.
	- - - - - - - - - - - - g per pot - - - - - - - - - - - -					
A Horizon						
Hosmer	1.80bc*	4.82abc	2.32bcd	1.36a	1.34ab	1.39ab
Iva	2.92ab	4.56abc	1.87bcd	1.58a	2.23a	1.67ab
Zanesville	2.79ab	4.10bcd	3.62b	1.30a	1.25ab	1.15ab
B Horizon						
Hosmer	1.10bc	3.70bcd	2.71bcd	0.30a	0.76ab	1.22ab
Iva	1.34bc	3.45cd	3.33bc	0.89a	1.26ab	0.68ab
Zanesville	0.82c	2.58d	2.14bcd	0.43a	0.70ab	1.34ab
Loess	1.14bc	5.30ab	1.73cd	0.50a	1.85a	0.93ab
Lacustrine	3.66a	6.05a	5.57a	0.96a	1.18ab	1.95a
Glacial Till						
Wisconsin	0.83c	3.71bcd	1.07d	0.02a	0.78ab	0.43ab
Unw. Ill.	1.25bc	2.49d	2.27bcd	0.57a	0.18b	0.19ab
Wx. Ill.	0.93c	4.03bcd	2.48bcd	0.88a	1.09ab	0.94ab
Blk. Fis. Shale	0.00c	0.00e	0.15e	0.00a	0.00b	0.00b
Gray Mar. Shale	0.48c	1.07e	1.80bcd	0.05a	0.08b	1.02ab
Brown Shale	1.73bc	4.74abc	1.76cd	0.35a	0.52ab	0.89ab
Sandstone	0.80c	2.52d	1.63cd	0.19a	0.28b	1.42ab

*Within columns, values among materials followed by the same letter are not significantly different at the 5% level. Within rows, values between treatments differing by more than 1.26 g for either species are significantly different.

available K and percent organic matter are similar to A horizons (Tables 6, 7, 8). It would appear that the underlying unconsolidated materials, the tills and loess, and the somewhat weathered brown shale are almost as productive as the B horizons. These materials generally are not statistically different in plant yield from the B horizons, and in several instances, produced higher yields; for example, wheat yields on sludge-treated brown shale and weathered Illinoian till and untreated brown shale. It is also apparent that gray and black shale are not likely to be productive even when amended with sludge and fertilizer.

Table 13 summarizes the analysis of variance in the greenhouse experiments. All of the main effects and two-way interactions were significant for plant yield which is apparent in Tables 11 and 12. The addition of amendments accenuated the material differences, and they were more apparent with wheat than alfalfa due to wheat's more rapid growth and its more consistent emergence.

The relationship among materials and the effects of treatments is easily seen for wheat yields in Figure 2. The three A horizons and three B horizons are represented by a single mean value to simplify comparisons. The addition of sludge vastly improved the growth on all materials except gray and black shale and was generally more effective than inorganic fertilizers. The sludge probably improved some of the physical properties as well as supplying nutrients. It appears that the addition of sludge to overburden material used in the rooting zone might be an attractive technique for field trials. The fairly uniform response of wheat on unamended glacial tills, loess, and B horizons became much more variable as well as improved when sludge was added.

The poor performance of the shales and sandstone was related, in part, to the slow and incomplete plant emergence reflected in Tables 14 and 15. Their adverse chemical and physical properties undoubtedly retarded emergence and growth. However, treatments with fertilizer or sludge did not consistently bring about improvement. Sludge reduced emergence and early growth of wheat when applied to unweathered Illinoian till and improved emergence on the brown shale; otherwise, it had little effect (Table 14). The addition of fertilizer caused only slight improvement in wheat and alfalfa on most materials (Table 14 and 15). In addition, surface crusting of weak structured, unconsolidated materials such as the glacial tills may have affected seed germination and rate of plant emergence. This appeared to be the case with alfalfa grown on unweathered Illinoian till when fertilizer and sludge were added.

The general ranking of overburden material for plant growth potential in greenhouse studies is lacustrine sediment $\leq$ A horizons > B horizons = glacial tills = loess $\leq$ brown shale > sandstone > gray shale > black fissile shale. Amendments ranked sewage sludge > fertilizer > control.

Elemental Content of Wheat Plants

Wheat plants harvested after 8 weeks growth in untreated (control) overburden materials and those amended with sewage sludge or N-P-K fertilizer plus lime were analyzed for elemental content (Table 16). Overburden materials in all treatments were watered as needed to maintain adequate moisture

TABLE 13. SUMMARY OF ANALYSIS OF VARIANCE FOR THE GREENHOUSE EXPERIMENTS.

Source of variation	Study #1	Study #2		
	Yield	Yield	Emergence	Height
	Significance*			
Overburden material	S	S	S	S
Treatment (Tr.)	S	S	S	S
Species (spp.)	S	S	NS	NS
Material X treatment	S	S	NS	NS
Material X species	S	S	NS	NS
Treatment X species	S	S	NS	NS
Material X Tr. X spp.	NS	S	NS	NS

*Tested at 5% level.

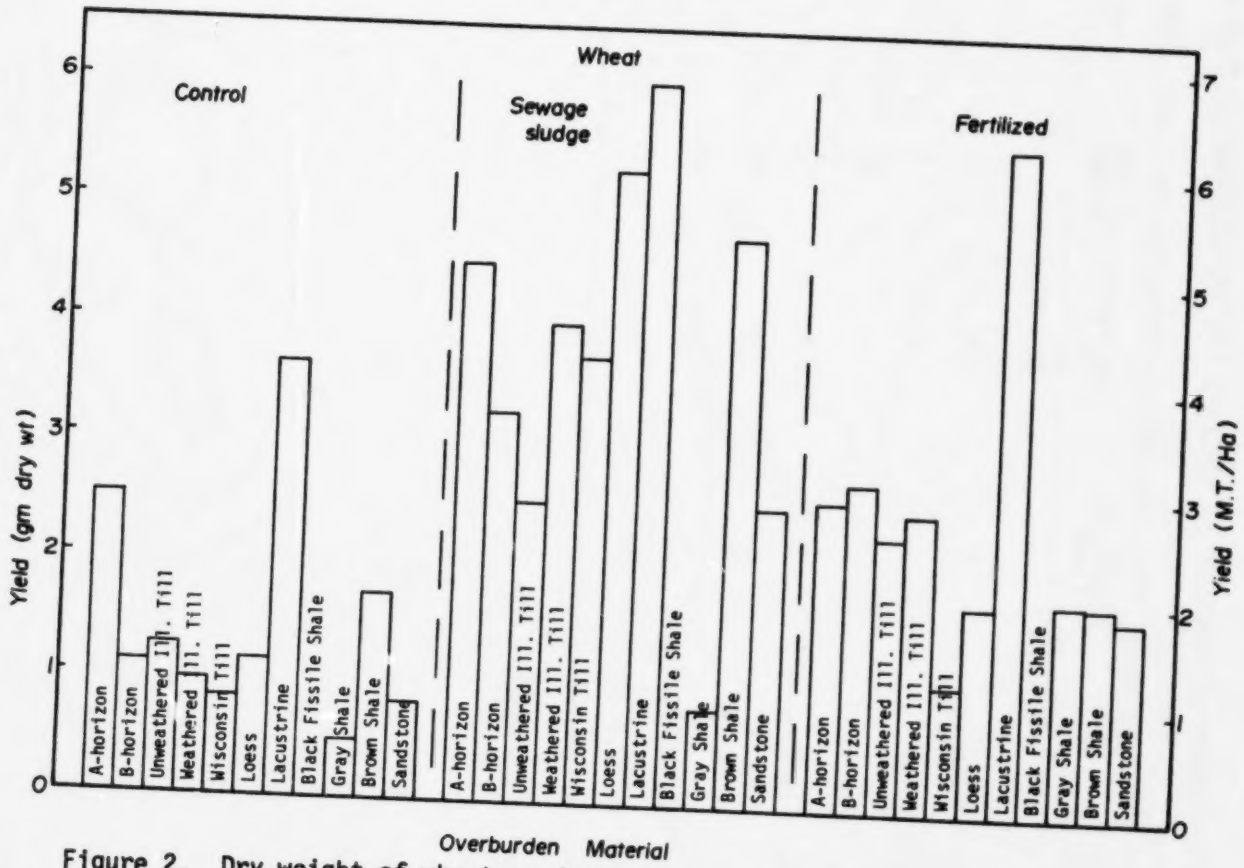

Figure 2. Dry weight of wheat produced in second greenhouse experiment as influenced by overburden material and treatment.

TABLE 14. EMERGENCE AND HEIGHT OF WHEAT PLANTS AFTER FOUR WEEKS GROWTH ON OVERBURDEN MATERIALS IN THE SECOND GREENHOUSE EXPERIMENT.

Overburden material	Treatment					
	Control		Sludge		Fertilizer	
	Emergence*	Height	Emergence	Height	Emergence	Height
	%	cm	%	cm	%	cm
A-Horizon						
Hosmer	85	18.0	85	20.0	80	21.0
Iva	75	16.0	75	16.5	80	20.0
Zanesville	90	18.5	85	20.0	85	21.0
A-Horizon Mean	83	17.5	82	18.8	82	20.7
B-Horizon						
Hosmer	70	19.5	85	21.5	90	22.0
Iva	85	15.5	65	16.0	85	16.5
Zanesville	80	19.5	85	20.5	85	19.5
B-Horizon Mean	78	18.2	78	19.3	87	19.3
Loess	80	17.0	90	16.5	90	17.5
Lacustrine	85	20.0	70	22.5	90	23.0
Glacial Till						
Wisconsin	85	15.0	65	14.0	85	17.5
Unw. Illinoian	80	17.0	20	5.5	90	19.0
Wx. Illinoian	75	16.5	75	15.5	85	20.5
Glacial Till Mean	80	16.2	53	11.7	87	19.0
Black Fissile Shale #1	0	0	5	10.0	25	12.0
Gray Marine Shale	40	8.0	35	6.0	50	9.0
Brown Shale	50	11.5	85	11.5	85	11.5
Sandstone	55	14.0	60	13.5	50	12.0

*Emergence percent is ratio of plants emerging/total seeds planted. Twenty seeds with germination capacity > 95% planted per pot.

TABLE 15. EMERGENCE AND HEIGHT OF ALFALFA PLANTS AFTER FOUR WEEKS GROWTH ON OVERBURDEN MATERIALS IN THE SECOND GREENHOUSE EXPERIMENT.

Overburden material	Treatment					
	Control		Sludge		Fertilizer	
	Emergence*	Height	Emergence	Height	Emergence	Height
	%	cm	%	cm	%	cm
A-Horizon						
Hosmer	95	14.0	90	14.5	95	16.5
Iva	70	10.0	80	13.5	70	16.0
Zanesville	80	15.5	75	13.0	75	14.0
A-Horizon Mean	82	13.2	82	13.7	80	15.5
B-Horizon						
Hosmer	75	15.0	75	14.5	75	15.5
Iva	65	11.0	80	15.5	70	14.5
Zanesville	80	14.5	70	14.0	65	15.5
B-Horizon Mean	73	13.5	75	14.7	70	15.2
Loess	60	11.5	85	14.0	80	15.0
Lacustrine	90	17.0	85	18.0	55	18.5
Glacial Till						
Wisconsin	45	8.0	0	0	55	11.5
Unw. Illinoian	55	8.5	40	4.0	15	8.0
Wx. Illinoian	55	10.0	80	13.5	70	13.0
Glacial Till Mean	52	8.8	40	7.2	47	10.8
Black Fissile Shale #1	30	7.5	0	0	20	3.5
Gray Marine Shale	50	7.5	25	5.0	45	7.0
Brown Shale	30	8.0	65	8.0	40	5.5
Sandstone	30	9.5	55	9.0	70	10.0

*Emergence percent is ratio of plants emerging/total seeds planted. Twenty seeds with germination capacity > 95% planted per pot.

supplies during seed germination and plant growth. Elements selected for analysis included P, Ca, Mg, Mn, Fe, Zn, B and Al. These were compared with interpretive standards for nutrient levels of wheat plants used by the Purdue Plant and Soil Analysis Laboratory. Due to instrument malfunction, K was not included in the analysis. For the black fissile shale, there was no yield of wheat in control and sludge treatments and less than 1/4 g per pot in the fertilizer treatment; therefore, no comparison of tissue elements was possible for this material. Likewise, wheat yield in some replicates of glacial till, shale and sandstone materials was too small for analysis, therefore, mean element content is based on incomplete or partial replication.

Phosphorus content of wheat tissue ranged from "low" to "medium", .11 to .28%, for plants grown on unamended control overburden materials. This is consistent with the very low and low levels of available P in the overburden itself for all materials except black fissile shale (Table 7). Amendment with sewage sludge and fertilizer resulted in minor increases in plant P, but not above "medium" levels, which are .21 to .50%, on Iva A and B horizon, loess, lacustrine and gray and brown shales. Phosphorus concentration in wheat plants grown on the three glacial tills was in the medium range for all treatments. Plant concentrations of calcium increased in response to both sludge and fertilizer treatments from "medium" to "high" levels in A and B horizon materials. Sludge alone resulted in higher Ca in plants grown on the tills, but little change was noted among treatments for other materials. Magnesium content of wheat on control treatments was "medium", .33 to .64%, for all materials except sandstone which was "high". Wheat magnesium levels remained essentially the same as the control, "medium" for nearly all materials, with sludge and fertilizer amendments.

Manganese levels in wheat tissue on overburden materials without amendment were "medium" to "high" for A horizon, Wisconsin and weathered Illinoian till and brown shale, and, "very high" for B horizon, loess, lacustrine, unweathered Illinoian till, gray shale and sandstone. Changes in foliar Mn due to sludge and fertilizer were erratic; however, there was some trend of increased Mn levels with sludge application for B horizon, loess, Wisconsin till and gray and brown shales and with fertilization on A horizon, loess and gray shale. Wheat iron content was "medium", 120 to 274 ppm, on all untreated overburden materials. Sludge treatment resulted in "high" levels of Fe for B horizon, loess and sandstone, but no practical change occurred in other materials. "Very high" levels of iron and aluminum occurred on two fertilized materials, B horizon and gray marine shale. In the case of the gray marine shale, this was probably due to the oxidation of S and resultant drop in pH. Zinc in wheat tissue on control treatments was "low" (20 ppm) for B horizon, "high" (103 ppm) for lacustrine and "medium" (26 to 68 ppm) on all other materials. Zinc content increased from "low" or "medium" to "high" levels in wheat on sludge-amended A horizon, B horizon, glacial tills and sandstone, while no major changes occurred for materials amended with fertilizer + lime.

Boron concentration in wheat tissue was "high" for plants grown on unamended gray marine shale and increased to "very high" levels with sludge and fertilizer treatments. This large accumulation in plant tissue is likely related to high extractable B in the gray marine shale (Table 7). Boron content was "medium" in wheat for all other materials and treatments.

TABLE 16. ELEMENTAL CONTENT OF WHEAT PLANTS GROWN EIGHT WEEKS IN SELECTED OVERBURDEN MATERIALS WITH AND WITHOUT AMENDMENTS IN POT CULTURE.

Overburden material	P			Ca			Mg			Mn		
	C*	S*	F*	C	S	F	C	S	F	C	S	F
	%			%			%			ppm		
Iva A-Horizon	.20	.26	.21	.96	1.27	1.01	.47	.54	.54	56	60	221
Iva B-Horizon	.11	.22	.28	.39	1.01	1.25	.33	.64	.60	426	455	196
Loess	.15	.25	.28	.58	.89	.88	.43	.75	.75	544	655	731
Lacustrine	.14	.15	.24	1.03	1.05	.82	.58	.57	.49	791	450	614
Wisconsin Till	.22	.31	.25	1.14	1.21	1.02	.60	.65	.49	94	442	86
Unw. Ill. Till	.22	.27	.23	.80	1.13	.83	.54	.62	.55	422	319	409
Wx. Ill. Till	.28	.24	.26	.84	1.15	.72	.60	.73	.54	332	232	182
Gray Marine Sh.	.13	.16	.30	.20	.27	.40	.49	.80	.83	445	733	601
Brown Shale	.18	.26	.27	.78	.71	.62	.64	.59	.54	142	351	130
Sandstone	.21	.18	.24	.54	.64	.49	1.19	1.39	.86	867	291	322

Overburden material	Fe			Zn			B			Al		
	C	S	F	C	S	F	C	S	F	C	S	F
	ppm			ppm			ppm			ppm		
Iva A-Horizon	168	162	238	53	73	62	12	12	10	200	166	288
Iva B-Horizon	151	332	563	20	87	67	7	14	22	122	357	622
Loess	120	361	268	43	69	53	22	14	17	124	302	219
Lacustrine	274	204	229	103	108	84	15	16	14	288	221	230
Wisconsin Till	212	297	208	56	75	52	16	12	13	250	281	202
Unw. Ill. Till	234	205	215	68	114	52	20	13	13	232	214	204
Wx. Ill. Till	222	196	165	56	89	53	16	10	7	258	285	202
Gray Marine Sh.	165	182	584	26	56	44	48	99	75	163	212	624
Brown Shale	202	197	244	62	61	40	11	16	13	226	252	337
Sandstone	144	338	258	59	88	41	21	18	15	174	454	296

*Treatments: C = Control, S = Sewage Sludge, F = Fertilizer.

The relationship between extractable B in unamended overburden materials and uptake of B in wheat, oats and alfalfa is shown in Figure 3. These data are combined means of B content in materials and plants used in greenhouse and outdoor container experiments. This comparison is of interest since gray shales, a very abundant material in the coal region, contain plant-available B sufficiently high (> 2 µg/g) to be toxic to sensitive crops (Reisenauer, Walsh and Hoeft, 1973). In addition, reduced plant growth was observed on gray shale that in part may be related to toxic accumulations of B. For all species, plants grown on gray shale contained more B than those grown on any other material. Concentrations of B in wheat and oats may have been sufficiently high to be toxic. Alfalfa, a luxury consumer of B, had higher B accumulations on all materials with the highest level occurring on gray shale; however, those levels are not considered toxic for alfalfa (Jones, 1972).

Aluminum content of wheat grown in unamended overburden was "medium" in A and B horizon, loess, gray shale and sandstone and "high" in lacustrine, the three glacial tills and brown shale. Plant Al increased to "high" or "very high" levels following sludge and fertilizer applications on B horizon, loess, gray shale and sandstone. Aluminum levels of 400^{+} ppm are considered "very high" and may exist at toxic levels. Although not included in Table 16, Cu levels were "medium" in wheat tissue for all overburden materials and treatments.

Based on elements analyzed, it appears that adequate concentrations are present in wheat plants grown on unamended overburden except for P which is low, .11 to .20%, in tissue for several materials. Element content of wheat generally increased with sludge and fertilizer amendments for most materials, but changes were somewhat erratic and in most materials not drastic. The "very high" to excessive tissue levels of Fe, Al and B on gray marine shale; Fe and Al on B horizon; and Al on sandstone may be potentially toxic to plants. Also, Mn concentrations > 351 ppm accumulating in wheat grown on one or more treatments of B horizon, less, lacustrine, unweathered Illinoian till, gray shale and sandstone are considered very high to excessive. Berg and Vogel (1968) observed Mn toxicity in the form of leaf margin chlorosis on several legume species grown in acid strip-mine spoils in Kentucky. Critical tissue concentrations were not determined, but Mn toxicity symptoms were most severe on materials with pH below 5.0

PLANT GROWTH STUDIES-OUTDOOR CONTAINERS

Ten overburden materials (Table 17) were collected from exposed highwalls at the Ayrcoe, Ayrshire, Chinook and Universal coal mines in October 1977. Material from the consolidated rock strata was partially fractured in extraction and placed directly in plastic-lined metal containers without further crushing. Thirty containers, three replications of each overburden strata, were arranged in three randomized blocks of 10 materials each at the Purdue University Martell Forest in Tippecanoe County, Indiana. Overburden materials did not receive soil amendments or supplemental irrigation.

Oats were planted in March and harvested in April, 1978, after 4 weeks

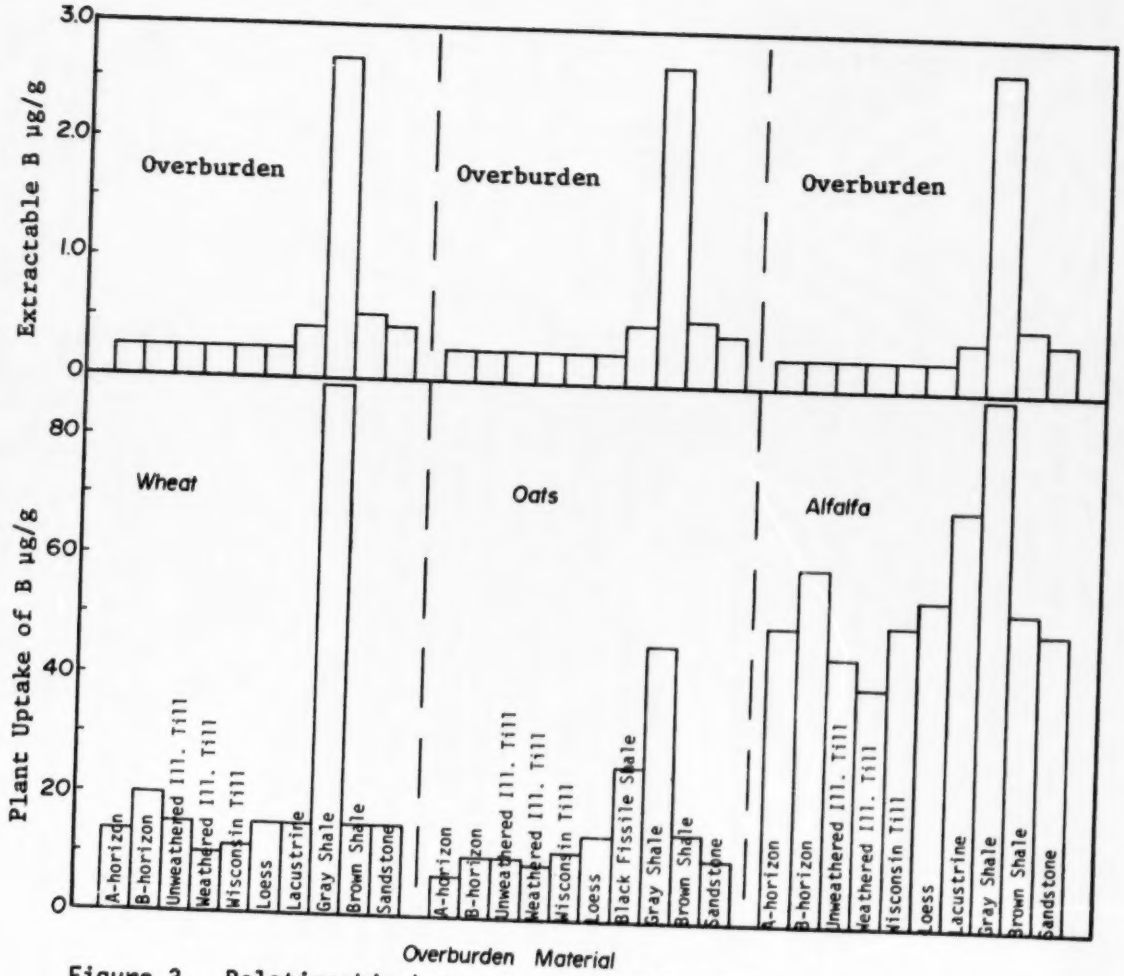

Figure 3. Relationship between extractable boron in overburden materials and uptake by plants.

TABLE 17. MEAN OAT YIELD, 4 WEEKS, AND TREE SURVIVAL AND GROWTH, 12 WEEKS, IN OUTDOOR CONTAINERS OF OVERBURDEN MATERIAL. EACH VALUE IS A MEAN OF THREE REPLICATES.

Overburden material	Oat yield dry wt.	Virginia Pine			Yellow-Poplar		
			Growth			Growth	
		Survival	Total height	Basal diameter	Survival	Total height	Basal diameter
	g	%	cm	cm	%	cm	cm
Iva A-Horizon	3.67a*	100	8.8a	0.18a	100	34.7a	0.41a
Iva B-Horizon	2.64b	100	8.1ab	0.19a	100	9.9b	0.19bc
Loess	3.13ab	100	8.9a	0.15ab	100	9.1b	0.12c
Unw. Illinoian Till	2.91ab	100	4.7c	0.11ab	100	4.3b	0.12c
Wx. Illinoian Till	3.13ab	100	5.8abc	0.14ab	100	6.7b	0.12c
Wisconsin Till	2.31bc	100	4.0c	0.12ab	100	10.8b	0.25b
Black Fissile Shale #1	0.57d	33†	0.7d	0.00c	0	0.0c	0.00d
Gray Marine Shale	1.56c	50	0.7d	0.05bc	0	0.0c	0.00d
Brown Shale	3.12ab	100	5.3bc	0.15ab	100	6.1b	0.20bc
Sandstone	2.52b	100	4.9bc	0.14ab	100	12.5b	0.15bc

*Means followed by the same letter are not significantly different at P = 0.05 according to Duncan's Multiple Range test.

†All tree seedlings were dead by December 1978, 7 months after planting.

of growth. Oat tissue was analyzed for 11 macro and micro nutrient elements. Two one-year-old seedlings of Virginia pine and yellow-poplar were planted in each container in April, 1978. Seedling total height and basal diameter were measured at time-of-planting and in August, 1978, after 12 weeks of growth. Seedling survival was determined in August and December, 1978. Leachate samples from the outdoor containers of overburden materials were collected periodically in 1978 and spring 1979 and analyzed for pH, soluble salts and nutrient element content. Seedling growth and leachate content will be monitored through the 1979 growing season.

Oats Yield

Mean oat yield after four weeks growth ranged from 3.67 to 0.57 g dry weight per container of 1461 cm^2 surface area, for the Iva A horizon and black fissile shale, respectively. Oat yields for the loess, unweathered and weathered Illinoian till, and brown shale averaged 14 to 21% less than the yield in A horizon, but these differences in mean yield were not significant at P = 0.05. Oat yield of 2.64 g in B horizon material was significantly less than in the A horizon, but was equivalent to yields in all other overburden materials except gray marine and black fissile shales. The poorest oat yield, 0.57 g, occurred in black fissile shale and next poorest yield, 1.56 g, in gray marine shale.

Oat vegetative tissue was analyzed for three macronutrients (P, Ca and Mg), five micronutrients (Fe, Mn, B, Cu and Zn) and three other minor elements (Na, Ba and Al) (Table 18). Due to the small sample size, less than 5 g dry weight oats tissue from each container, analysis for other elements was not feasible. Nutrient element concentration in oat tissue was compared to interpretive standards of very low, low, medium, high and very high levels used by the Purdue University Plant and Soil Analysis Laboratory.

Phosphorus and calcium contents of 0.23 to 0.46% and 0.24 to 0.66%, respectively, in oat tissue for all overburden materials were in the "medium" level. The highest P accumulation occurred in plants grown in brown shale and weathered Illinoian till and was not related to soil test levels, which were all very low. The highest Ca content in tissue was associated with A horizon and Wisconsin till which were both well supplied with Ca (Table 7). Magnesium concentrations ranging from 0.11 to 0.30% were in the "low" to "medium" plant tissue levels with the highest content occurring on B horizon which also had the highest exchangeable Mg (Table 7) and lowest on gray marine shale. Of the micronutrients, Cu and Zn oat tissue concentrations were in the "medium" range with the highest levels of both elements occurring on loess. Boron and Mn levels in tissue were "medium" for oats grown in most overburden materials. Gray marine shale contained a high level of water extractable B and produced oats with 46 ppm, "high" level, in the tissue. The loess contained only a moderate amount of extractable Mn, yet produced oats with the highest level, 208 ppm. The plant Fe content was adequate, "medium", for all materials except loess and brown shale where accumulations of 555 and 673 ppm were excessive and possibly in the toxic range. Likewise, Al accumulations were excessively high and probably at toxic levels for plants grown in loess and brown shale. This is unexpected since the exchangeable Al levels were not high nor were the pH's low. Aluminum was "medium" to "high" for oats grown

TABLE 18. MEAN ELEMENT CONTENT OF OAT PLANTS GROWN FOUR WEEKS IN OUTDOOR CONTAINERS OF OVERBURDEN MATERIAL. EACH VALUE IS A MEAN OF THREE REPLICATES.

Overburden material	Element										
	P	Ca	Mg	Na	Mn	Fe	Cu	Zn	B	Al	Ba
	%				ppm						
Iva A-Horizon	0.29c*	0.66a	0.15e	0.02e	63d	111b	23b	31c	7e	91b	4cd
Iva B-Horizon	0.26c	0.48bc	0.30a	0.28b	189a	215b	18b	34c	10cde	205b	7c
Loess	0.30c	0.54b	0.26bc	0.15cde	208a	555a	32a	49a	14cd	835a	25a
Unw. Illinoian Till	0.23c	0.49b	0.23d	0.03e	128bc	184b	19b	32c	10cde	141b	2cd
Wx. Illinoian Till	0.37b	0.40cd	0.28ab	0.42a	118bc	161b	22b	38bc	9de	204b	5cd
Wisconsin Till	0.29c	0.65a	0.25cd	0.11de	78cd	178b	20b	33c	12cde	137b	1d
Black Fissile Shale #1	0.24c	0.46bc	0.24cd	0.12de	104bc	98b	20b	34c	26b	98b	2cd
Gray Marine Shale	0.31c	0.25e	0.11f	0.31ab	93bcd	169b	26ab	32c	46a	235b	3cd
Brown Shale	0.46a	0.24e	0.27abc	0.19bcd	134b	673a	24b	39bc	15c	1024a	15b
Sandstone	0.25c	0.34d	0.27abc	0.25bc	101bcd	175b	21b	45ab	11cde	351b	2cd

*Means in columns followed by the same letter are not significantly different at P = 0.05 according to Duncan's Multiple Range Test.

in all other overburden materials. The tissue content of Na and Ba appeared to be within the normal range for most materials. Overall, it appears that tissue element concentrations were adequate or normal for most elements analyzed. However, deficiencies may exist in B for plants grown in all materials except gray marine shale and in Mg for plants grown in gray marine shale and A horizon material. Jones (1972) and Brady (1974) indicate that plant B concentrations of less than 15 ppm is considered deficient. It is probable that toxic levels of Al and Fe accumulated in plants grown in loess and brown shale.

Tree Seedlings

Survival of Virginia pine and yellow-poplar seedlings after one growing season, August 1978, was 100% in all overburden materials except black fissile and gray marine shales (Table 17). Virginia pine survival was 33 and 50% in black fissile and gray marine shale, respectively; while, survival of yellow-poplar was zero in these materials. By December 1978, all Virginia pine in black fissile shale were dead and no change in survival was noted for both species in all other materials.

Virginia pine height growth of 5.8 to 8.9 cm in 12 weeks was superior in A and B horizon soil, loess and weathered Illinoian till. The poorest growth, consistent with low survival, occurred in the black fissile and gray marine shales. Virginia pine basal diameter growth was generally slight with little variation among overburden materials except for significantly lower growth in black fissile and gray marine shale.

Height and basal diameter growth of yellow-poplar, 34.7 cm and 0.41 cm, respectively, in Iva A horizon were significantly better than all other materials. No net growth was observed on yellow-poplar in black fissile and gray marine shales and all seedlings were dead by August 1978. Growth of yellow-poplar was not significantly different in all other overburden materials.

Under conditions of this outdoor container study and based on first-year growth characteristics of oats, Virginia pine and yellow-poplar, it is evident that the black fissile and gray marine shales without amendments are unsuitable as a plant growth medium. The A horizon soil provides the best overall plant growth potential. Other materials studied appear to support adequate plant growth and may be suitable media alone or in mixture in the rooting zone. The excessive plant accumulations of Fe and Al from loess and brown shale did not appear to affect the short-term growth evaluated in outdoor containers.

Leachate Analyses

Chemical analysis of leachate samples from outdoor containers of overburden materials are reported for the May 1978 and April 1979 collection periods (Table 19). Leachate was available from only a few of the coarse materials in summer 1978 and therefore was not summarized since all materials could not be compared. Leachate analysis was limited to pH, electrical conductivity and four essential nutrient elements.

TABLE 19. CHEMICAL ANALYSIS OF LEACHATE COLLECTED IN MAY 1978 AND APRIL 1979 FROM OUTDOOR CONTAINERS OF OVERBURDEN MATERIAL. EACH VLAUE IS A MEAN OF THREE REPLICATES.

Overburden material	pH		Elect. Cond.		P		K		Ca		Mg
	5/78	4/79	5/78	4/79	5/78	4/79	5/78	4/79	5/78	4/79	4/79
			mmhos/cm		- - - - - - - - - - - - ppm - - - - - - - - - - - -						
Iva A-Horizon	7.7	7.3abc*	0.44	0.50b	0.05	0.00	2.6	4.0b	67	99bc	15c
Iva B-Horizon	6.9	6.9abc	0.23	0.20b	0.05	0.00	0.6	0.4b	20	17d	11c
Loess	6.8	6.1bc	0.12	0.12b	0.05	0.00	0.5	0.4b	12	15d	6c
Unw. Illinoian Till	8.0	7.8a	1.97	0.89b	0.07	0.00	11.5	4.6b	240	146b	55c
Wx. Illinoian Till	7.0	6.8abc	0.23	0.17b	0.07	0.00	0.3	0.2b	18	13d	8c
Wisconsin Till	7.6	7.9a	0.32	0.35b	0.15	0.00	4.0	0.7b	45	61cd	17c
Black Fissile Shale #1	7.5	3.6d	2.37	5.30a	0.03	0.43	16.7	8.1b	300	367a	2067a
Gray Marine Shale	7.3	5.7c	5.35	4.80a	0.05	1.27	29.2	11.6b	360	377a	1480b
Brown Shale	8.1	6.7abc	0.21	0.15b	0.00	0.05	72.0	78.3a	23	12d	11c
Sandstone	7.8	7.5ab	0.38	0.37b	0.13	0.00	25.5	1.1b	18	22d	15c

*Means in columns followed by the same letter are not significantly different at P = 0.05 according to Duncan's Multiple Range Test.

Leachate from all materials was slightly acid to alkaline, pH 6.8 to 8.1, in May 1978. By April 1979, there was a decrease in pH for leachate from all materials except Wisconsin till and B horizon soil. A major decrease in pH occurred in leachate from black fissile, gray marine and brown shales. This is likely due to rapid weathering of these soft shales and the formation of acid as the exposed S compounds, probably pyrites, oxidized. Calcium and magnesium were especially high in leachate from the black fissile and gray marine shales and moderately high from unweathered Illinoian till.

Phosphorus in the leachate was negligible from all overburden materials except black fissile and gray marine shales in April 1979. Moderate levels of potassium, 25 to 72 ppm, occurred in leachate from the gray marine and brown shales and sandstone in May 1978; however, by April 1979 only leachate from brown shale had significant potassium content. Electrical conductivity exceeded 1 mmhos/cm in leachate from unweathered Illinoian till, black fissile shale and gray marine shale in May 1978. In April 1979, electrical condictivity of leachate was excessive, 4.8 to 5.3 mmhos/cm, for only the gray marine and black fissile shales. This high level of soluble salts in the soil solution could be detrimental to seed germination and plant growth.

Chemical properties of overburden materials used in the outdoor containers (Table 20) varied somewhat from those used in greenhouse studies (Tables 7 and 8). The material was collected from the same mines and strata but at different locations and therefore further reflects variability encountered. The major differences occured in the rock strata. The pH was less acidic, 6.5 versus 3.7, in black fissile shale and more alkaline, 8.1 versus 5.5, in sandstone at time-of-collection in October 1977 as compared with previous samples. After one year of weathering, April 1979, the pH of material in the surface 2 inches of outdoor containers decreased, with the most dramatic adjustment occurring in the black fissile and gray marine shales (Table 20). Electrical conductivity, organic matter and P content were lower in the black fissile shale, gray marine shale and sandstone; and, K content was higher in all shales and sandstone than material collected for previous studies.

PREDICTION OF PLANT GROWTH POTENTIAL

It is apparent when plant growth in greenhouse and outdoor container experiments (Tables 11, 12 and 17) are compared with physical and chemical overburden properties (Tables 6, 7 and 8) that no single property is a reliable predictor of plant growth. Variability in individual overburden properties within strata accounts for some of the difficulty in identifying specific predictors. However, the occurrence of properties known to cause severe plant toxicity, suppressed growth or mortality in a given material can be identified and used to rule out that material as plant growth media. This is evident in the case of the black fissile and gray shales where some critical or limiting factors occur at extreme levels. Certain individual easily measured factors such as pH, electrical conductivity, potential acidity or soil texture may be adequate to eliminate the material from consideration if the tolerance range is known for given plant species.

TABLE 20. CHEMICAL PROPERTIES OF OVERBURDEN MATERIALS USED IN OUTDOOR CONTAINERS.

Overburden materials	pH		Electric conduc.	Organic matter	P	K
	10/77	4/79				
			mmhos/cm	%	---kg/ha---	
Iva A-Horizon	7.0	6.6	.15	1.0	11	81
Iva B-Horizon	5.8	5.5	.33	0.4	7	246
Loess	5.7	5.2	.03	0.2	11	103
Unw. Illinoian Till	8.1	7.7	.65	0.4	2	83
Wx. Illinoian Till	6.9	6.8	.12	0.2	11	123
Wisconsin Till	8.1	7.7	.13	0.5	3	83
Black Fissile Shale #1	6.5	2.7	2.80	3.3	6	358
Gray Marine Shale	6.2	3.0	3.80	3.4	7	616
Brown Shale	7.0	7.0	.24	0.5	6	314
Sandstone	8.1	7.7	.39	0.2	2	166

Plant yields from greenhouse studies and corresponding overburden properties were examined by step-wise regression techniques. The results (Table 21) indicate the limited usefulness of such an approach when single factors exhibit overriding importance in some materials and are unimportant in others. This may lead to fortuitous correlations that are unrelated to cause-and-effect. When the overburden properties (Tables 6, 7 and 8) and greenhouse yield data (Tables 11 and 12) are evaluated in accord with regression results, it becomes apparent that some properties normally considered desirable (e.g. high levels of K, P, Ca, Mg and CEC) occur in the same materials that produce low yields because of very low pH or other detrimental factors. This results in the favorable properties disappearing from the regression equation.

Electrical conductivity of the soil solution and water storage capacity were the properties that occurred most frequently as the best single factor in the regressions (Table 21). Manganese, magnesium and bulk density were the best factors in some equations. The negative regression of Mg with plant yield is unexpected in this region and is due to the occurrence of high Mg in a few overburden materials that were very undesirable in other respects. Potassium, B, Al, pH, P and % clay appear as additional factors in the multiple correlation. The relationship of these factors with yield was in the expected direction with the exception of P which was negative instead of positive as one would anticipate. The explanation for the negative correlation of P is similar to that for Mg.

The single factors alone were poor to moderate predictors of plant growth, R^2 = .39 to .68, and the addition of one or two other factors in step-wise regression gave only modest improvement. It appears that measurement of only those properties listed in these equations (Table 21) would not allow adequate prediction of plant growth response. However, it is obvious that these properties should be examined along with others indicated in previous studies and in agriculture and forestry practice.

TABLE 21. OVERBURDEN PROPERTIES MOST CLOSELY CORRELATED BY STEP-WISE REGRESSION TO PLANT YIELD IN GREENHOUSE EXPERIMENTS.

Experiment Treatments Species	Best single factor		Multiple correlation	
	Factor*	R^2	Factor(s)*	R^2
Experiment 1				
Control				
Alfalfa	-EC†	.39	-EC	.39
Wheat	-Mg	.42	-Mg, +K	.76
Fertilizer				
Alfalfa	-EC	.68	-EC, -B	.80
Wheat	-Mn	.50	-Mn, -Mg	.67
Experiment 2				
Control				
Alfalfa	-BD	.47	-BD, -Al, -Clay	.71
Wheat	$+H_2O$	.42	$+H_2O$, +pH	.58
Fertilizer				
Alfalfa	$+H_2O$	.56	$+H_2O$	.56
Wheat	$+H_2O$	.51	$+H_2O$, -P	.62
Sludge				
Alfalfa	-BD	.46	-BD, +pH	.62
Wheat	-EC	.58	-EC, -B	.67

*Factor symbols: EC, electrical conductivity of soil solution; Mg, exchangeable Mg; K, exchangeable K; Mn, extractable Mn; B, extractable B; BD, bulk density of repacked materials; Al, exchangeable Al; Clay, percent clay; H_2O, percent water by volume between 1/3 and 15 bars; P, available P by Bray P1 extraction; pH, 1:1 soil to water by volume.

†Sign indicates positive or negative correlation with yield.

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APPENDIX A

LABORATORY PROCEDURES SYNOPSIS

1. Elemental Analyses

Phosphorus: Bray No. 1, Fiske-Subbarrow Method. Use 1.5 g of soil in 15 ml of 0.025 NH_4Cl in .03 N NH_4F. Shake 5 min at 200 opm. Filter. Take a 5 ml aliquot, add 2 ml acid ammonium molybdate solution and mix. Add 2 ml P-C agent (Aminonaphthol-sulfonic acid) and mix. Develop color. Read between 15 min - 45 min at 600 mμ. Compare with standards.

Potassium: 1.5 g soil in 15 ml of 1 N ammonium acetate, pH 7.0. Shake 5 min at 200 opm. Filter. Read on AA. Compare with standards.

Calcium and Magnesium: To 1.5 g soil add 15 ml 1 N ammonium acetate pH 7.0. Shake 5 min at 200 opm and filter. Dilute with Lanthanum Chloride (1500 ppm). Read on AA Spectrophotometer. Compare with standards.

Sodium: To 5 g soil add 15 ml 1 N ammonium acetate pH 7.0. Shake 5 min at 200 opm. Do not filter. Read supernatant liquid on AA Spectrophotometer. Compare with standards.

Aluminum: Leach 10 g of soil with five 20 ml aliquots of 1 N KCl. Add 10 drops of 0.1% phenolphthalein to filtrate. Titrate the filtrate to a permanent pink endpoint using standardized 0.1 N NaOH. Add more indicator, if necessary, as some may be precipitated with the $Al(OH)_3$. Record base. Add one drop of 0.1 N HCl to bring the filtrate back to colorless. Add 10 ml of 4% to filtrate and stir. If the filtrate contains exchangeable Al, the pink color returns. Stirring constantly, titrate the filtrate with 0.1 N HCl until color disappears. Add 1 or 2 drops of indicator. If color returns, titrate until no color returns for two minutes. The meq. of acid used is equal to the meq. of exchangeable Al.

Manganese: 10 g soil are extracted with 50 ml 0.1 N phosphoric acid, shake 15 min, filter 20 ml, add 2 ml of conc. H_3PO_4, evaporate. Organic matter is oxidized with H_2O_2. Add distilled water and 0.3 g of trisodium periodote. Heat 20 min. Dilute to exactly 20 ml with distilled water. Read color on Coleman Model 8 Photo-Electric Colorimeter at 540 mμ.

Boron: Add 40 ml of nearboiling distilled H_2O to 20 g of soil and reflux 5 min. Add 2 drops of 20% $Cacl_2$ solution, cool, and filter through boron-free filter paper. Take 0.5 ml aliquot of the soil.

extract, add 2 ml of curcumin reagent and mix thoroughly. Evaporate to dryness in a 55± 3 C water bath or oven. Heat 15 min, dissolve in 10 ml of 95% ethyl alcohol and transfer to a colorimeter tube. Allow any turbidity present to settle out. Read in colorimeter within 2 hours at 540 mμ. Evaluate against a standard curve.

Molybdenum; Extraction is made with pH 3.3 $NH_4C_2O_4$ using a 10:1 dilution factor. Extract is centrifuged and analyzed directly with atomic absorption.

Sulfur: The Leco Sulfur Analyzer is used. Ground samples are treated with combustion accelerations (iron and tin) and heated to ca 1,600 C in a stream of purified O_2. The high temperature is generated by using an induction furnace to indice an electrical field in the accelator-treated sample. The SO_2 liberated is collected in dilute HCl containing potassium iodide, starch and trace amounts of potassium iodate. This is titrated automatically with KIO_3 through the use of a photocell to maintain the initial optical density of the blue color formed by reaction of the starch with the iodine formed by reaction of KIO_3 and KI in the presence of HCl.

2. Other Chemical

pH: 1:1 soil to water volume. Mix 5 seconds. Let stand 10 min. Stir, read pH with pH meter.

Buffer pH: Add 10 ml of SMP buffer solution (pH 7.5). Shake at 200 opm for 10 min. Let stand 30 min. Stir and read pH.

Cation Exchange Capacity:

Extractable acidity. To 10 g of air dry soil add 100 ml of extraction solution ($BaCl_2$, triethanolamine). Stopper, shake well, and let stand overnight. Run blank. Filter, wash with extracting solution. Bring up to 250 ml. Add indicator (bromcresol green and methyl red) and titrate with 0.2 N HCl to pink c[illegible]nge end-point. Calculate.

Base extraction. Use macerated paper in 60 ml fritted glass funnels. Extract with a total of 100 ml 1 N ammonium acetate pH 7.0 (allow to sit overnight in 30 ml extracting solution). Run Ca, Mg, K, and Na on extract. Calculate C.E.C. and report in meq. Calculate percent base saturation.

Organic Matter: Oxidize with potassium dichromate and conc. sulfuric acid by standing for 30 min; add water, phosphoric acid, sodium fluoride, and diphenylamine indicator. Titrate with 0.5 N ferrous ammonium sulfate.

Soluble Salts: To 25 g of air-dry soil add 50 ml of distilled water and shake well for 1 min. Cover and let sit for 30 minutes. Filter into test tube. Determine K-value of unknowns after standardizing solubridge with 0.01 N KCl set at 141.

Potential Acidity: FeS_2 is completely oxidized with H_2O_2 and the product, H_2SO_4, is measured titrametrically with 0.01 N NaOH.

3. Physical Analyses

Particle Size: Samples are sieved to remove particles larger than 2 mm. The remaining sample is dispersed with sodium hexametaphosphate, placed in settling containers and subsampled at appropriate intervals with a pipette. The subsample is dried at 105 C and weighed.

Moisture Retention: Two samples of disturbed overburden were measured. Moisture retained at 15 bar tension was measured by placing about 10 g of material passing a 5 mm sieve in a pressure membrane apparatus. Fifteen bars of pressure was applied and the sample was allowed to equilibrate. The sample was removed, weighed, dried at 105 C, and weighed again. Moisture retained at 1/3 bar tension was measured by placing loose, unsieved material in metal cans (8 cm diameter x 5 cm deep, small hole in bottom) and wetting the material to saturation. Saturated samples were placed in a pressure plate apparatus, put under 1/3 bar pressure and allowed to equilibrate. The samples were then removed, weighed, dried at at 105 C, and weighed again.

Bulk Density: Determined using 8 cm x 5 cm metal cans. Loose samples were placed in the cans, wet to saturation, dried at 105 C and weighed.